MWH
MONTGOMERY WATSON HARZA

November 1, 2002

✓ KA.
11/14/02

Kevin Adler
Remedial Project Manager
Region V, Mail Code SR-6J
U.S. Environmental Protection Agency
77 West Jackson Boulevard
Chicago, Illinois 60604-3590

RE: Work Plan for Phase 3 Investigation
Oxygen Release Compound (ORC) Pilot Study
American Chemical Service NPL Site RD/RA

Dear Mr. Adler:

The *South Area ORC Pilot Study Evaluation* report was submitted to the Agencies in September 2002. It reported positive results from the ORC applications but also recommended further investigation and sampling in order to develop final recommendations for a remedial approach to groundwater impacts at the site. This Work Plan provides details of the recommended supplemental investigation. It has been prepared by MWH to be used in conjunction with the Quality Assurance Project Plan (QAPP), Field Sampling Plan (FSP), and Site Safety Plan (SSP) which the U.S. Environmental Protection Agency and Indiana Department of Environmental Management (IDEM) approved. The following three investigative tasks are planned:

- Task 1 – Collect additional groundwater samples from MW06, ORCPZ102, and ORCPZ103 during the 4th quarter 2002;

- Task 2 – Collect groundwater samples using a direct-push technology (DPT) rig from the area around ORC Array 5; and

- Task 3 – Collect several aquifer matrix (soil) samples by DPT rig from near/beneath Colfax Avenue and the pipeline.

Task 1 – Additional Groundwater Sampling

Groundwater samples were collected from a number of wells in the South ORC Pilot Study Area during the 3rd quarter and analyzed for volatile organic compounds VOCs. Additional samples from three of these wells MW06, ORCPZ102, and ORCPZ103 will be useful in evaluating the affects of the ORC applied during the pilot study and in assessing the chemical environment of the upper aquifer in the south area.

Another round of groundwater sampling is planned for the fourth quarter. The locations, number of samples and analytical parameters are summarized in Table 1. VOC results will be used to extend the trend analyses developed during the pilot study. The inorganic analyses including dissolved organic carbon (DOC), chemical oxygen demand (COD), and iron will be used to provide an indication of the chemical environment in the potential source area and groundwater plume. The locations of the wells are shown on Figure 1. Samples will be collected in accordance with the groundwater sampling SOP in the approved Long-Term Groundwater Monitoring Plan (LTGMP) (September 2002).

Task 2 – DPT Groundwater Sampling Activities
In order to identify the appropriate remedial approach to the groundwater plume, it is essential to understand the source mechanism that has fed the plume. There is still some uncertainty regarding the nature of the source in the South Area and therefore additional sampling is planned to better characterize the area of highest concentration at the upgradient end of the plume. The plan for this task is to use direct push technology (DPT) to collect one-time groundwater samples from a number of locations in between the existing monitoring wells.

Groundwater samples will be collected using a DPT rig from within and immediately surrounding ORC Array 5, located northeast of the Colfax Avenue – Reder Road intersection (Figure 2). This is the location where highest benzene concentrations have been detected in previous investigations. The total VOC concentrations detected by the Tracer Investigation in 1996 are shown on Figure 1.

During the pilot study, 10 pounds of ORC were applied for each one-foot thickness of the aquifer in Array 5 in a grid with 10-foot spacing. To evaluate residual effects from that application, groundwater samples will be collected from six locations between the monitoring wells in this area by DPT. Table 2 summarizes the number of samples to be collected and lists the analytical parameters for each sample. Figure 2 shows the proposed locations for the DPT groundwater samples. These sampling points are located along the general groundwater flow path through the ORC application areas. Two samples will be collected north of the house, one sample upgradient of Array 5, two samples within Array 5, and one sample downgradient of the array.

These samples will be collected following the general practice outlined in the groundwater standard operating procedure (SOP) in the LTGMP. The DPT rig will push down to the middle portion of the saturated zone in the upper aquifer (between 20 to 25 feet below ground surface). The first DPT rod will contain a two to three foot long stainless steel screen. After insertion to the appropriate depth, the DPT rods will be pulled back to expose the screen to the saturated zone. A sample of groundwater will then be collected following low-flow sampling protocol.

The equipment used for collecting the groundwater sample includes a peristaltic pump and dedicated tubing. The inlet of the tubing will be set within the screened section of DPT rods. Groundwater will be purged and parameters recorded until the parameters stabilize, as defined in the LTGMP SOP. A sample will be collected at each location after stabilization. All DPT rods, screens, and equipment will be decontaminated between each sample. Clean, unused tubing will be used at each separate sampling point.

Task 3 – DPT Soil Sampling Activities

There may be organic compounds in the aquifer matrix which are not detectable in groundwater samples, yet which have the potential to act as a source, and may have an effect on the remedial approach selected. Therefore, this task has been included in the investigation to collect aquifer matrix materials and analyze them for potential contaminants. Previous investigations suggest that the origin of the south area groundwater plume in the vicinity of the intersection of Colfax Avenue and Reder Road. The existence of the road and buried utilities along it and also the presence of the high-pressure pipeline in this area have limited sampling at this location in the past.

Locations for aquifer matrix sampling have been selected to provide soil matrix material that is representative of the upper aquifer in the vicinity of this potential source area. A total of eight DPT borings will be drilled and up to 12 samples will be collected around the pipeline and Colfax Avenue intersection. Each sample will be analyzed for a range of parameters, summarized in Table 3. The results will be used to evaluate the potential that residual organic material trapped in the aquifer matrix may act as a secondary source of groundwater contamination and/or that it may act to interfere with or limit the effectiveness of certain remedial approaches. The locations of the eight proposed soil borings are shown in Figure 3.

The DPT rig will collect soil samples in four-foot long acetate sleeves. Upon retrieval, each sleeve representing the upper aquifer will be cut open exposing a four-foot section of the aquifer matrix. The soil will be screened using a photo-ionization detector (PID). A sample from the section of the aquifer yielding the highest PID readings and/or visible evidence of contamination will be collected for laboratory analysis.

If there are no elevated PID readings, the sample will be collected from the interval representing the water table. If elevated PID readings occur at an interval other than at the water table, both the water table interval and the elevated PID reading interval will be sampled (at up to four DPT borings). After the samples for laboratory analysis have been collected, the soil from each sampling location will be classified using the United Soil Classification System (USCS) to the termination of the boring.

Quality Assurance/Quality Control Procedures

Quality Assurance/Quality Control (QA/QC) procedures will be performed in general accordance with the U.S. EPA-approved QAPP. All samples will be analyzed at CompuChem Laboratories in Cary, North Carolina. Sample handling and chain-of-custody procedures will be conducted in accordance with the procedures outlined in the QAPP to ensure that sample integrity is maintained.

Quality Control (QC) samples will be prepared for all VOC analyses (water and soil). Quality control sample frequency will follow the procedures outlined in the QAPP: one trip blank to accompany each VOC sample shipment, one duplicate sample and one equipment blank sample per ten groundwater samples, and one matrix spike/matrix spike duplicate (MS/MSD) sample per 20 samples. (QC samples are summarized in the tables.)

Laboratory analysis and data validation procedures for VOC analyses in groundwater will follow the QAPP procedures and protocols. All other groundwater analyses (i.e. natural attenuation parameters) will be checked in accordance with the laboratory's general QA/QC procedures. No formal validation will be completed for these analyses.

Laboratory analysis and data validation procedures for VOC analyses in soil will be conducted under the guidelines provided in Attachment 1. This attachment provides the laboratory standard operating procedures for analysis of VOCs in soils and the quality control and data validation guidelines for the soil VOC analyses.

Reporting
Upon completion of these supplemental field activities, a report will be prepared to summarize the field procedures and the sampling results. The report will make recommendations and be submitted to the U.S. EPA and IDEM for review and comment.

Schedule
The tentative schedule of the activities outlined in this work plan is:

- November 2002 - Collect DPT groundwater and soil data (Task 2 and 3).
- December 2002 – Collect 4^{th} quarter groundwater samples (Task 1)
- January-February 2003 – Compile and analyze data.
- March 2003 – Submit report to Agencies.

If you have any further questions, please do not hesitate to contact me at (630) 836-8923.

Sincerely,

MWH

Peter J. Vagt, CPG, Ph.D.
Vice President

Attachments: Table 1. Monitoring Well Re-sampling Summary
Table 2. DPT Groundwater Sampling Summary
Table 3. DPT Aquifer Matrix Sampling Summary
Figure 1. South Area 1996 Tracer Sampling Results
Figure 2. Proposed DPT Groundwater Sampling Locations

Figure 3. Proposed DPT Aquifer Matrix Sampling Locations
Attachment 1. Laboratory SOPs and QC information

cc: P. Kasarabada, IDEM
B. Magel, Karaganis, White, and Magel
L. Campbell, Black & Veatch

CAS/cas/PJV/jmf
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2090601.010901

TABLES

TABLE 1.
Monitoring Well Re-Sampling Summary
Phase 3 Investigation
American Chemical Service NPL Site
Griffith, Indiana

Task 1 Groundwater Analyses	VOCs	Diesel Range Organics	Gasoline Range Organics	Dissolved Organic Carbon	Chemical Oxygen Demand	Nitrate	Nitrite	Ammonia-N	Total Kjedahl Nitrogen	Total Iron and Manganese	Dissolved Iron and Manganese	Sulfate	Methane, Ethane, Ethene
Analytical Method	SW8260B	SW8015B	SW8015B	EPA 415.1	EPA 410	EPA 300.0	EPA 300.0	EPA 350.1	EPA 351.3	SW6010B	SW6010B	EPA 300.0	RSK 175
4th Quarter 2002													
MW06	1	1	1	1	1	1	1	1	1	1	1	1	1
ORCPZ102	1	1	1	1	1	1	1	1	1	1	1	1	1
ORCPZ103	1	1	1	1	1	1	1	1	1	1	1	1	1
QA/QC													
Duplicates	1												
Matrix Spike	1												
Matrix Spike Duplicate	1												
Eqipment blank	2												
Trip Blanks	1												
TOTAL	**9**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**

Notes
Analytical Methods with the prefix "SW" indicate SW-846 Methods.
Total iron and manganese analyses will be unfiltered, whereas dissolved iron and manganese samples will be filtered using disposable .45 micron filters.
Two equipment blanks will be collected since two different pumps will be used to sample the three wells.

TABLE 2.
DPT Groundwater Sampling Summary
Phase 3 Investigation
American Chemical Service NPL Site
Griffith, Indiana

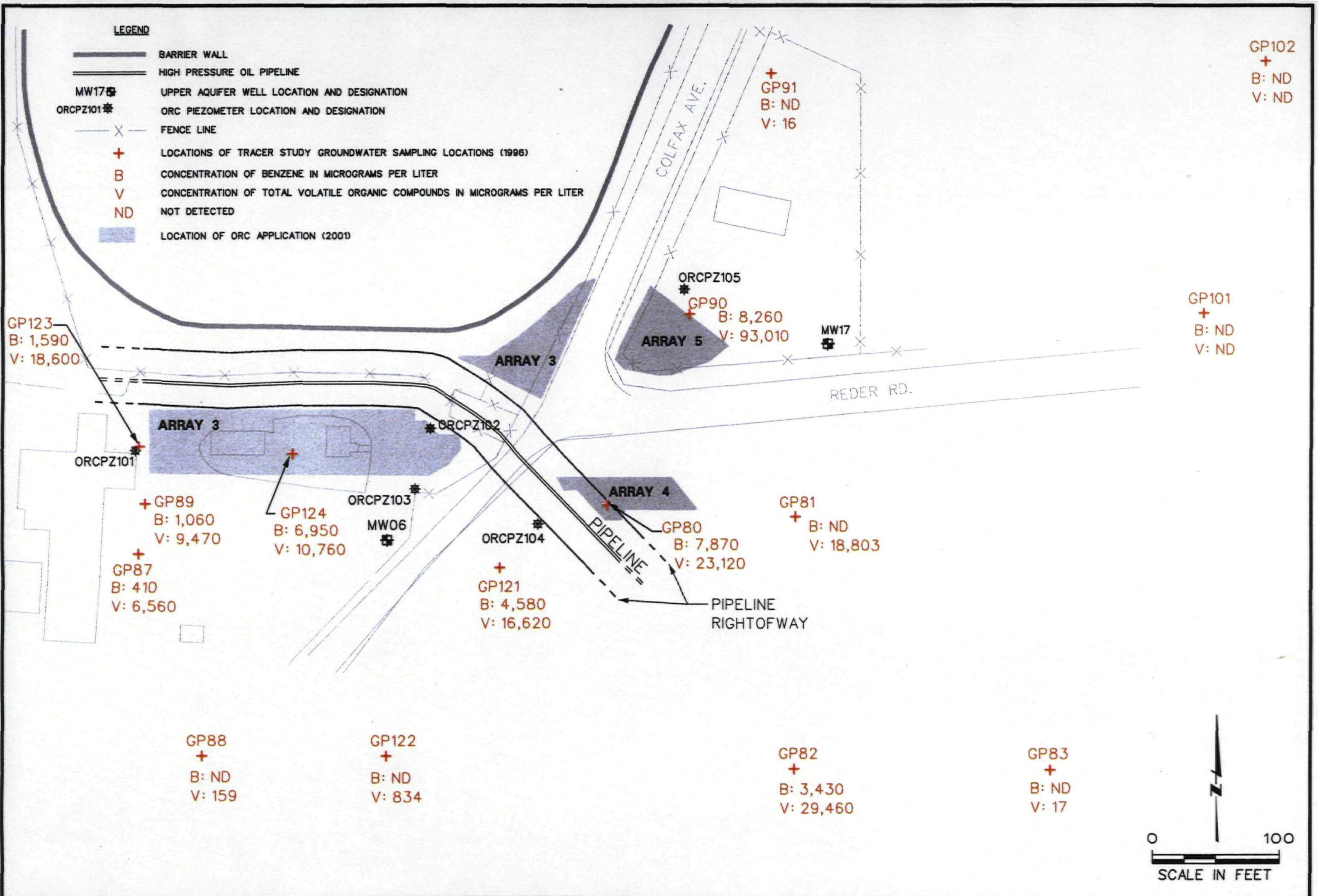
Task 2 Groundwater Analyses	VOCs	Diesel Range Organics	Gasoline Range Organics	Dissolved Organic Carbon	Chemical Oxygen Demand	Nitrate	Nitrite	Ammonia-N	Total Kjedahl Nitrogen	Total Iron and Manganese	Dissolved Iron and Manganese	Sulfate	Methane
Analytical Method	SW8260B	SW8015B	SW8015B	EPA 415.1	EPA 410	EPA 300.0	EPA 300.0	EPA 350.1	EPA 351.3	SW6010B	SW6010B	EPA 300.0	RSK 175
Task 2 - DPT Groundwater Samples													
Up to Six locations	6	6	6	6	6	6	6	6	6	6	6	6	6
QA/QC													
Duplicates	1												
Matrix Spike	1												
Matrix Spike Duplicate	1												
Equipment blank	1												
Trip Blanks	1												
TOTAL	11	6	6	6	6	6	6	6	6	6	6	6	6

Notes
Analytical Methods with the prefix "SW" indicate SW-846 Methods.
Total iron and manganese analyses will be unfiltered, whereas dissolved iron and manganese samples will be filtered using disposable .45 micron filters.
The equipment blank will consist of sampling distilled water through an unused section of tubing.

TABLE 3.
DPT Aquifer Matrix Sampling Summary
Phase 3 Investigation
American Chemical Service NPL Site
Griffith, Indiana

Task 3 Soil Analyses	VOCs	Diesel Range Organics	Gasoline Range Organics	Total Organic Carbon	Chemical Oxygen Demand
Analytical Method	**SW8260B/5035**	**SW8015B**	**SW8015B**	**EPA 415.1**	**EPA 410**
Task 3 - DPT Soil Samples					
Up to 8 locations	12	12	12	12	12
QA/QC					
Duplicates	3				
Matrix Spike	1				
Matrix Spike Duplicate	1				
TOTAL	**17**	**12**	**12**	**12**	**12**

Notes
Analytical Methods with the prefix "SW" indicate SW-846 Methods.



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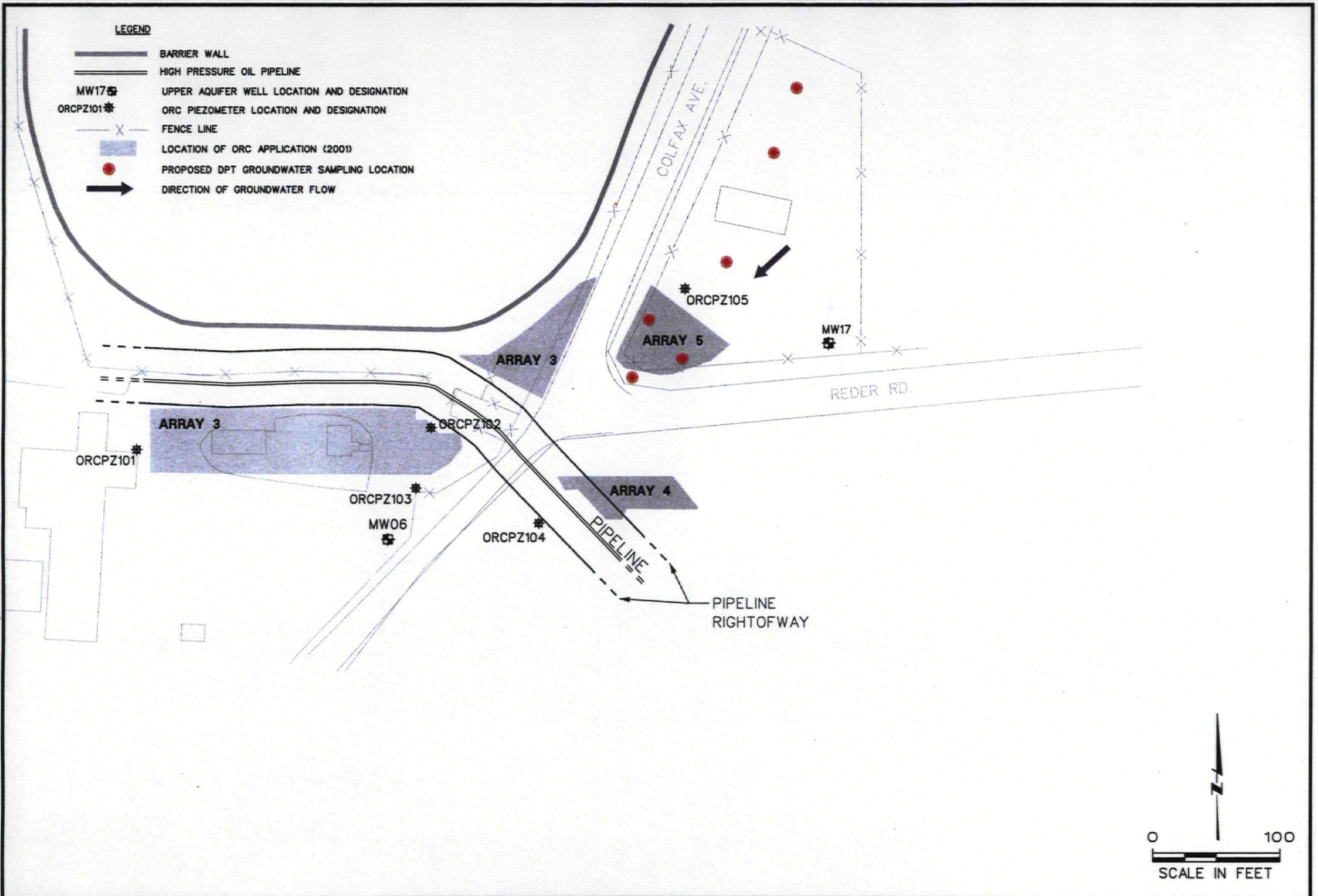
SOUTH AREA 1996 TRACER SAMPLING RESULTS

FIGURE 1

Plot Date: 31-OCT-2002
File: J:\209\0601 ACS\0109 ORC_South\Drawings\PHASE 3 FIG 1.dgn
Job No: 0000000



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PROPOSED DPT GROUNDWATER SAMPLING LOCATIONS

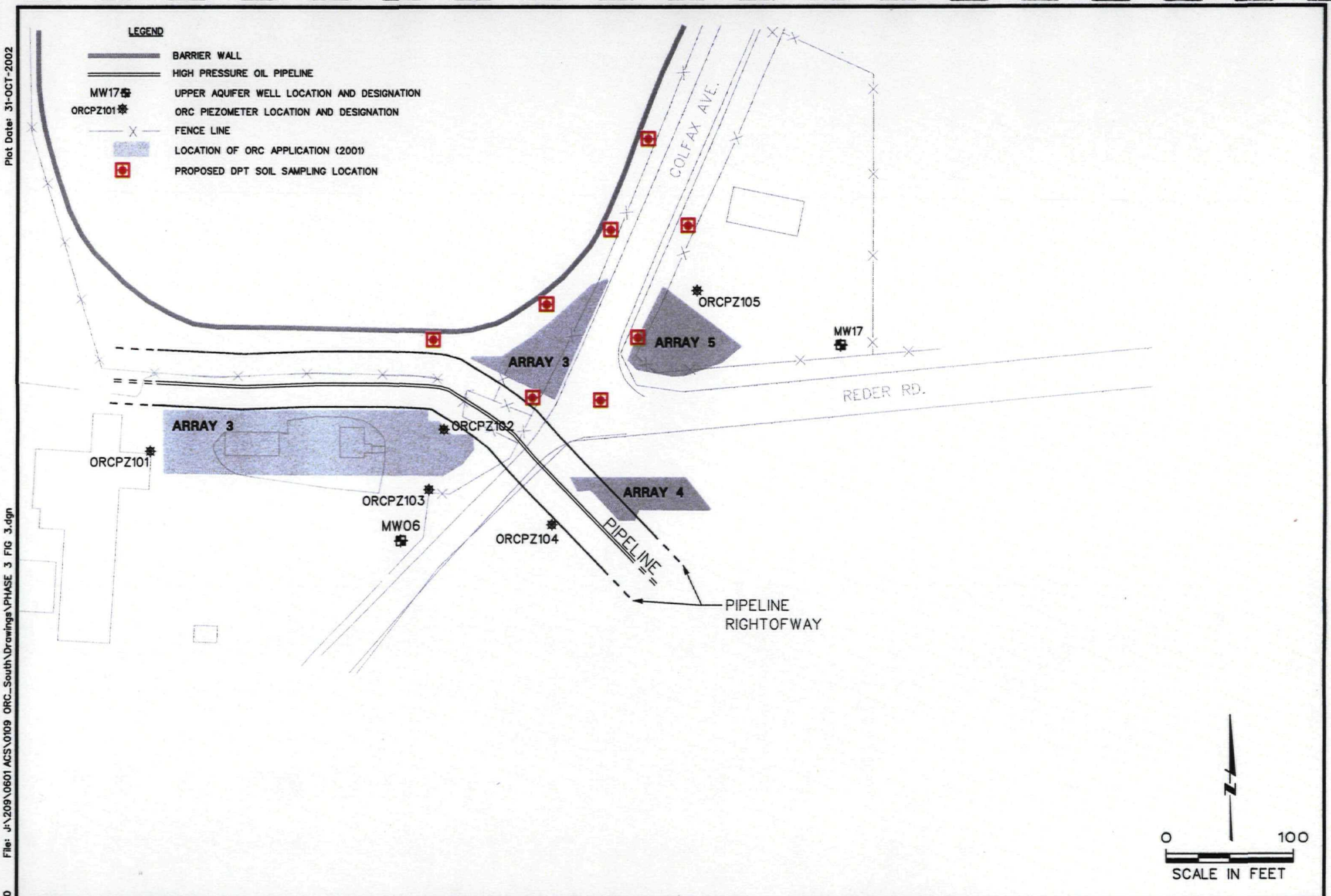
FIGURE 2

Plot Date: 31-OCT-2002
File: J:\209\0601 ACS\0109 ORC_South\Drawings\PHASE 3 FIG 2.dgn
Job No: 0000000

Plot Date: 31-OCT-2002

File: J:\209\0601 ACS\0109 ORC_South\Drawings\PHASE 3 FIG 3.dgn

Job No: 0000000



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PROPOSED DPT AQUIFER MATRIX SAMPLING LOCATIONS

FIGURE 3

1

Table A-1
Analytes, Practical Reporting Limits, and Maximum Contaminant Levels
American Chemical Service, Griffith, Indiana

Soil					
	Compuchem Standard		U.S. EPA SSL Commercial/Industrial Scenario		
Target Parameter	PQL (µg/kg)	MDL (µg/kg)	Ingestion-Dermal (mg/kg)	Inhalation (mg/kg)	Migration to Groundwater (DAF=20) (mg/kg)
VOCs by SW8260B/5035					
1,1,1-Trichloroethane	10.0	0.57	NE	1,200	2.0
1,1,2,2-Tetrachloroethane	10.0	0.27	16	1	0.003[(1)]
1,1,2-Trichloroethane	10.0	0.49	56	2	0.02
1,1-Dichloroethane	10.0	0.44	110,000	1,700	23
1,1-Dichloroethene	10.0	0.58	5	0.1	0.06
1,2-Dichloroethane	10.0	0.34	35	0.6	0.02
1,2-Dichloropropane	10.0	0.27	47	21	0.03
2-Butanone (MEK)	10.0	1.77	NE	NE	NE
2-Hexanone	10.0	1.12	NE	NE	NE
4-Methyl-2-pentanone (MIBK)	10.0	1.23	NE	NE	NE
Acetone	10.0	4.29	110,000	NE	16
Benzene	10.0	0.30	58	1	0.03
Bromodichloromethane	10.0	0.29	51	NE	0.6
Bromoform	10.0	0.58	400	88	0.8
Bromomethane	10.0	0.48	NE	NE	NE
Carbon Disulfide	10.0	0.71	110,000	720	32
Carbon Tetrachloride	10.0	0.62	24	0.6	0.07
Chlorobenzene	10.0	0.30	23,000	180	1
Chloroethane	10.0	0.63	NE	NE	NE
Chloroform	10.0	0.32	520	0.5	0.6
Chloromethane	10.0	0.28	NE	NE	NE
cis-1,2-Dichloroethene	10.0	0.33	11,000	NE	0.4
cis-1,3-Dichloropropene	10.0	0.34	NE	NE	NE
Dibromochloromethane	10.0	0.37	NE	NE	NE
Ethylbenzene	10.0	0.40	110,000	400	13
Methylene chloride	10.0	0.46	420	22	0.02
Styrene	10.0	0.30	230,000	1,500	4
Tetrachloroethene	10.0	0.62	61	18	0.06
Toluene	10.0	0.57	230,000	650	12
trans-1,2-Dichloroethene	10.0	0.60	23,000	NE	0.7
trans-1,3-Dichloropropene	10.0	0.34	NE	NE	NE
Trichloroethene	10.0	0.46	290	8	0.06
Vinyl chloride	10.0	0.49	4	1	0.01
Xylenes (total)	10.0	1.25	1,000,000	NE	190
1,2,4-Trichlorobenzene	10.0	0.10	6,800	3,200	5
1,2-Dichlorobenzene	10.0	0.25	62,000	600	17
1,3-Dichlorobenzene	10.0	10	NE	NE	NE
1,4-Dichlorobenzene	10.0	0.32	80	NE	2

Table A-1
Analytes, Practical Reporting Limits, and Maximum Contaminant Levels
American Chemical Service, Griffith, Indiana

Notes:
Analyte list is derived from CLP criteria
PQL Practical Quantiation Limits from Compuchem and Air Toxics
MDL Method Detection Limits from Compuchem and Air Toxics
U.S. EPA United States Environmental Protection Agency
SSL - Soil Screening Levels, EPA Document Number: EPA540/R-96/018 (July 1996)
DAF Dilution Attenuation Factor
N/A Not Applicable
NE Not Established
(1) The PQLs for this parameter is greater than the generic SSL. For this parameter, the sample data will be reported to the MDL. Sample results greater than the MDL but lower than the PQL will be reported as estimated concentrations (flagged), and all non-detections will be reported as less than the MDL.

Table A-2
Quality Control Acceptance Criteria for Volatile Organic Compounds by SW846 8260B
American Chemical Service, Griffith, Indiana

Analyte	Soil LCS (% Rec)	Soil MS/MSD (% Rec)	Soil MS/MSD (% RPD)
VOCs by SW-846 8260B/5035			
1,1-Dichloroethene	75-138	59-172	≤22
Benzene	75-129	66-142	≤21
Chlorobenzene	78-122	78-122	≤21
Toluene	76-119	59-139	≤21
Trichloroethene	75-121	62-137	≤24
Surrogates:			
Dibromofluoromethane	33-150	33-150	N/A
Toluene-d8	55-125	55-125	N/A
4-Bromofluorobenzene	46-150	46-150	N/A
1,2-DCA-d4	43-145	43-145	N/A

Notes:
N/A Not Applicable
Rec Recovery
VOCs Volatile Organic Compounds
LCS Laboratory control sample
MS/MSD Matrix spike/matrix spike duplicate
RPD Relative percent difference
Limits from CompuChem

Table A-3
Volatile Organic Compounds Gas Chromatography/Mass Spectrometry – Method SW846 8260B
Calibration Specification and Corrective Action Summary
American Chemical Service, Griffith, Indiana

Analytical Method[a]	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry	Volatile Organic Compounds	Tune instrument with a 4-bromofluorobenzene standard (BFB)	Every 12 hours	• Must meet key ions and ion abundance criteria established by method (Refer to Table 4-4)	• Retune instrument • Repeat standard analysis
		Initial multi-point calibration; 5 point minimum. Lowest point at or below PQL. Includes calibration check compounds (CCC) and system performance check compounds (SPCC), and Internal Standards Compounds (IS).	Prior to analysis, and as required	• RSD< 30 % for CCC; Average RF ≥ 0.1 for SPCC (≥0.3 for chlorobenzene, 1,1,2,2-Tetrachloroethane) • If mean % RSD for all compounds < 15%, average RF may be used; linear calibration required	• Evaluate system • Repeat calibration
		Continuing calibration verification (CCV): CCC, SPCC, and IS	Every 12 hours	• Percent difference (%D) <20% for CCC; RF ≥ 0.1 for SPCC (≥0.3 for chlorobenzene and 1,1,2,2-Tetrachloroethane). • Mean % D for all compounds < 20%. • Retention time for each internal standard must be within 30 seconds of most recent CCV and the EICP area for all internal standards must be within –50% to +100% of the most recent CCV.	• Evaluate system/standard • Reanalyze calibration check standard • Repeat initial calibration
		Method blank	1 per preparation batch (≤ samples)	• < Method Detection Limit	• Reanalyze method blank • Recalibrate
		Internal standards	Every sample, method blank, LCS, and MS/MSD	• Retention time for each internal standard must be within 30 seconds of most recent CCV and the EICP area for all internal standards must be within –50% to +100% of the most recent CCV	• Evaluate system/standard • Reanalyze samples • If still out, report both sets of data • Narrate all outliers

Table A-3
Volatile Organic Compounds Gas Chromatography/Mass Spectrometry – Method SW846 8260B
Calibration Specification and Corrective Action Summary
American Chemical Service, Griffith, Indiana
(continued)

Analytical Method[a]	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (continued)	Volatile Organic Compounds	Surrogate spike	Every sample, method blank, LCS, MS/MSD	• Surrogate recoveries within QC acceptance criteria. (Refer to Table 4-2) • One surrogate may be out if > 10% recovery	• Reanalyze sample one • If still out, report both sets of data • Narrate all outliers
		Matrix spike (MS)	1 per preparation batch (≤20 samples)	• Percent recovery within QC acceptance criteria (Refer to Table 4-2)	• If LCS and surrogate recoveries are acceptable and MSD also outside criteria consistent with MS, problem may be attributed to matrix interference • Reprep/reanalyze both MS and MSD. • Narrate all outliers
		Matrix spike duplicate (MSD)	1 per preparation batch (≤20 samples)	• % Recovery and/or RPD within QC acceptance criteria (Refer to Table 4-2)	• Same as MS
		Laboratory control sample (LCS)	1 per preparation batch (≤20 samples)	• % Recovery within QC acceptance criteria (Refer to Table 4-2)	• Reanalyze LCS • Reprep/reanalyze LCS and all associated samples • Narrate all outliers

Notes:
EICP Extracted ion current profile
QC Quality control
RF Response factor
RPD Relative percent difference
RSD Relative Standard Deviation
(a) EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), (U.S. EPA Third Edition, Final Update III, December 1996).

Table A-4
4-Bromofluorobenzene (BFB) Mass Intensity Criteria
American Chemical Service, Griffith, Indiana

Mass	Required Intensity (relative abundance)
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base peak, 100% relative abundance
96	5 to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5 to 9% of mass 174
176	Greater than 95%, but less than 101% of mass 174
177	5 to 9% of mass 176

EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846), (U.S. EPA Third Edition, Final Update III, December 1996).

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501 Madison Avenue
Cary, NC 27513

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure ______ revised procedure ✓ outdated procedure (archive) ______

◆ Procedure Code: IP 481B SOP Section #: 1.3.2.4 Revision #: 5

SOP Title: GC/MS Analysis of Low Concentration Volatiles in Soil/Sediment/Sludge Samples by SW846 and NYSASP

Effective date: (QA fills in) 1/14/02

◆ Procedure prepared by: Linda Carter

Date: 1/10/02

◆ Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign)

Date: 011402

◆ Reason for change: Addition of statistical control limits & change in NCDENR duplicate frequency

◆ This procedure meets the requirements of the following approved method references: SW846, 3rd Edition, Update III, 12/96 Methods 8260B and 5035; New York State Analytical Services Protocol, NYSASP, June 2000

Procedure approved by Quality Assurance Representative:
(Not needed if signed above)

Date:

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: ______________________ Date: __________

Annual Review—Signature: ______________________ Date: __________

Annual Review—Signature: ______________________ Date: __________

Instrument Procedure 481B: GC/MS Analysis of Low Concentration Volatiles in Soil/Sediment/Sludge Samples by SW-846 and NYSASP

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Instrument Procedure 481B: GC/MS Analysis of Low Concentration Volatiles in Soil/Sediment/Sludge Samples by SW-846 and NYSASP

1.0 Scope and Application

This is a general purpose **procedure** for the identification and simultaneous measurement of purgeable volatile organic compounds in a variety of solid matrices following Method 8260B **and incorporating Method 5035.** The method is applicable to a wide range of organic compounds. Target compounds that may be analyzed by this method are listed in Table 1, Attachment 1, along with their associated internal standards and quantitation ions. Note, however, that many of these compounds are not routinely analyzed.

Method detection limits (MDL) and reporting limits are shown in Attachment 3.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Low concentration level volatiles in soil/sediment/sludge samples are analyzed using a closed system purge and trap technique (Method 5035). Field samples are collected in vials containing a preservative solution (sodium bisulfate), immediately sealed and shipped to the laboratory at 4°C ± 2°C. Alternatively, an EnCore sampling device (or equivalent) is used to obtain a 5g or 25g sample that does not contain headspace. When the EnCore device is used, it is sent refrigerated to the laboratory and, within 48 hours of sampling ,a weighed aliquot must be transferred to a vial containing a sodium bisulfate solution.

A special autosampler is used for the analysis. This device allows the sample vial to remain closed while reagent water and a solution containing internal standards and surrogates are injected by a needle through the septum. The device allows the mixture in the sample vial to be stirred, using a magnetic stir bar. The needle used to pierce the septum to deliver the water, internal standards, and surrogates is then the source of an inert gas which is introduced at the top of the sample vial. The same needle has entrance holes located above the sample/water level to collect and transfer the headspace onto a sorbent column, where the purgeables are trapped. The autosampler also provides for heating the sample vial to 40°C while the contents are being stirred and the sample constituents purged and trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatograph (GC) wide-bore capillary column. The GC is temperature-programmed to separate the purgeables that are then detected with a mass spectrometer (MS).

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

If the low level standard concentration is not at least three times higher than the MDL value, the standard concentration is adjusted upward in order to achieve this minimal ratio. It may be adjusted higher than three times depending on the concentration range of the calibration curve and the ability to meet method linearity requirements. An exception to this is for CLP methods where the MDL is only required to be lower than the reporting limit.

The reporting limit for CLP is the Contract Required Quantitation Limit (CRQL) for organics and the Contract Required Detection Limit (CRDL) for inorganics.

3.3 Reporting Units – ug/kg

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each **7** calendar day period during which field samples in a case are received **(14** calendar days **if requested by the client)** beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

4.0 Interferences

4.1 Impurities in the purge gas or methanol, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. Gas lines from the gas tanks to the instrument must be either stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used. When potential interfering peaks are noted in laboratory method blanks, it may be necessary to reduce solvent contamination in the laboratory, purge the methanol used to prepare standard solutions, purge the reagent water with helium or nitrogen, change the purge gas source, or regenerate the molecular sieve purge gas filter.

4.2 Samples can be contaminated by diffusion of purgeable organics (particularly methylene chloride, fluorocarbons, and other common laboratory solvents) through the septum seal into the sample during storage and handling. Therefore, these samples are stored in GC/MS VOA laboratory refrigerator #2; separate from laboratory standards, and they must be analyzed in a room in which the atmosphere is demonstrated to be free of all potential contaminants that will interfere with the analysis. Because methylene chloride will permeate PTFE tubing, all GC carrier gas lines and purge gas plumbing are to be constructed from stainless steel or copper tubing.

4.3 Contamination by carryover can occur whenever a sample is analyzed after a sample that contains high levels of organic compounds. Whenever an unusually concentrated sample is encountered, it must either be followed by analysis of an instrument blank or the next sample must be closely monitored to check for cross-contamination. For samples containing large amounts of water soluble materials, high boiling compounds, or high purgeable levels, it may be necessary to clean the purge and trap apparatus by purging a 10-20% methanol solution, followed by baking the purge and trap apparatus and the analysis of an instrument blank to confirm that the system is free from contamination. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4.4 Instrument Problems/Preventative Maintenance

4.4.1 If a low response is observed for the early eluting compounds such as the gases, replacement of the trap or septum may be necessary. In addition, adjustments to the purge flow may be necessary to achieve a desired response for these compounds. If such adjustments do not help, it may be necessary to check the fittings on the purge and trap device and on the column for leaks. This is done with a helium leak detector and certain software utility programs.

4.4.2 Column maintenance or replacement may be necessary if peak tailing or broad chromatographic peaks are observed.

5.0 Safety

5.1 The toxicity and carcinogenicity of many chemicals used in this method have not been precisely determined: each chemical should be treated as a potential health hazard. Exposure to these chemicals should be minimized. Preparation of calibration standards, blanks, and samples is performed in a fume hood to minimize risk.

5.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,2-dichloroethane, 1,1,2,2-tetrachloroethane, 1,1,2- trichloroethane, chloroform, 1,2-dibromoethane, trichloroethene, and vinyl chloride.

5.3 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.4 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 Syringes

6.1.1 5-mL Hamilton gastight syringe with Luerlock tip

6.1.2 10-mL Hamilton gastight syringe with Luerlock tip
6.1.3 25-mL Hamilton gastight syringe with Luerlock tip

6.1.4 10-µl Hamilton syringe

6.1.5 1-µl Hamilton syringe

6.2 Volumetric Flasks and Pipets

6.2.1 Assorted volumetric flasks ranging from 50-mL to 1000-mL

6.2.2 10-mL graduated pipet in 1/10-mL graduations

6.3 Vials

6.3.1 40 mL screw-top, PTFE-lined, septum-sealed vials, each containing a magnetic stirring bar.

6.4 Analytical Column

6.4.1 J&W Scientific DB624

6.4.1.1 30 m or 75 m, 0.53-mm ID (internal diameter) with 3.0-µm film thickness

6.4.2 Supelco SPB-624 75-m Megabore column

6.4.2.1 75 m, 0.53-mm ID with 3.0-µm film thickness

6.4.3 J&W DB 624

6.4.3.1 60 m, 0.32 mm ID, 1.8 µl film

6.4.4 Restek RTX-624

6.4.4.1 60 m, 0.32 mm ID, 1.8 µl film

6.5 Mass Spectrometer (MS)

6.5.1 The MS scans 35-300 amu at 0.7-sec scan time in the electron impact mode at 70 eV (nominal).

6.5.2 Hewlett Packard 5890 GC

6.5.3 Hewlett Packard 6890 GC

6.5.4 Hewlett Packard 5972 MSD

6.5.5 Finnigan INCOS 500 mass spectrometers

6.6 Interface (GC to MS)

6.6.1 Type:

6.6.1.1 Jet separator

6.6.1.2 Direct capillary interface

6.6.2 Temperature: 250°C

6.6.3 Alternate: column direct to MS

6.7 Data System

6.7.1 A computer is interfaced to the mass spectrometer to allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.7.2 The data processing software searches any GC/MS data file for ions of specified mass and plots abundance versus time or scan numbers. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software integrates the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, the software compares sample spectra against reference library spectra. The reference library used is the NIST Mass Spectral Library.

6.7.3 For data acquisition, The INCOS 500 systems use Prolab software on Pentium computers.

6.7.4 For data acquisition, the Hewlett Packard systems use ChemStation software on Pentium computers.

6.7.5 For data processing, the Hewlett Packard HP 9000 series 735 Unix Workstation employing HP Chemserver with Target3 and Envision software by Thru-Put Systems is used.

6.8 Gas Chromatograph

6.8.1 Varian 3400 conditions are listed below:

- Carrier Gas — Helium
- GC mode — Capillary
- Injection Port Temp — 225°C
- Interface Temperature — 225°
- Initial Temperature — 0°C
- Final Temperature — 185°C
- Column Flow Rate — 25 ml/minute
  (10 ml/minute column flow +15 ml/minute makeup gas

6.8.2 The listed column flow rate is approximate. Flow rate is adjusted to optimize linear velocity of an unretained compound (butane) through the column. Optimum linear velocity for the column is 30 - 45 cm/second.

6.8.3 GC temperature program

6.8.3.1 This GC program is provided as an example; parameters may vary depending on the equipment.

- 0°C - 0°C for 2 minutes
- 0°C - 105°C @ 7°C/minute
- 105°C-185°C @ 26.7°C/minute
- 185°C-185°C
- Hold until all compounds elute.

6.9 Data Storage

6.9.1 Magnetic tape storage device: The magnetic tape storage device is capable of recording data and is suitable for long-term, off-line storage.

6.10 Purge and Trap Autosampler System

6.9.1 Tekmar LSC

6.9.1.1 Tekmar LSC 2000 or LSC 3000 with glass frit bottom liquid sample purging vessel and Luerlock valve.

6.9.1.2 The Tekmar LSC 2000 may be equipped with a Tekmar ALS 2016 autosampler for aqueous samples.

6.9.1.4 The absorption trap must be at least 25 cm long and have an internal diameter of at least 0.105 inches (0.2667 cm).

6.9.2 Archon ALS

6.9.2.1 The Archon Model 5100, 4552 Purge and trap autosampler interfaces directly to a Tekmar 3000 Purge and Trap Concentrator.

6.9.2.2 The autosampler is designed for soil samples and utilizes 40 ml VOA vials with low bleed Teflon septa.

6.9.2.3 The Archon ALS has the capacity of up to 51 vials.

6.10.2 Trap Packing

6.10.2.1 Supelco "K" Trap

6.10.2.2 Carbopak B

6.10.2.3 Carboxen 1000 & 1001

7.0 <u>Reagents and Standards</u>

Standards are prepared by the Organic Standards chemist. Details for the preparation are contained in the standard operating procedures (SOP) for that area (Section 7.0 of the SOP collection.) Standards are stored separately from samples at –10 to -20° C in the laboratory when not in use. Fresh standards are prepared on a weekly basis.

7.1 Reagent Water-All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Methanol (B&J Scientific, purge and trap grade)

7.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent

7.4 Tuning Standard

7.4.1 Bromofluorobenzene - Standard ID# 7008 at 25 µg/mL. Two µl are injected onto the column every 12 hours.

7.5 Calibration Standards

7.5.1 For the initial calibration, the internal standard solution is added automatically by the Archon Purge and Trap Autosampler. For all subsequent analyses, both the internal standard and the surrogate solutions are added automatically by the Archon autosampler.

Table 2: Standard Preparation. Units for column header values are µg/kg.

Std. ID	010	020	050	(100)	150*	200
8260 I.S.**	1.0	1.0	1.0	1.0	1.0	1.0
8260 S.S.	1.0	2.0	5.0	10.0	15.0	20.0
8260 Mix #1	0.5	1.0	2.5	5.0	7.5	10.0
8260 Gases	0.5	1.0	2.5	5.0	7.5	10.0
8260 Mix #2	0.5	1.0	2.5	5.0	7.5	10.0
8260 Mix #2B	0.5	1.0	2.5	5.0	7.5	10.0
8260 Mix #3A	0.5	1.0	2.5	5.0	7.5	10.0
8260 Mix #4A	0.25	0.5	1.25	2.5	3.75	5.0
8260 Mix #4C	0.5	1.0	2.5	5.0	7.5	10.0
8260 Mix #5	0.25	0.5	1.25	2.5	3.75	5.0
8260 Mix #6	0.25	0.5	1.25	2.5	3.75	5.0
8260 Mix #7	0.5	1.0	2.5	5.0	7.5	10.0

*This standard level only used with a 6-point initial calibration
( ) Continuing calibration verification concentration

7.5.1.1 The standard concentration presented on table header are for the compounds in 8260 Mix #1, the gases, 8260 Mix #2, 8260 Mix #2B, Mix #4C.

7.5.1.2 The standard concentrations for compounds in 8260 Mix #3A are 10, 20, 50, 100, 150, and 200 µg/kg with THF at 40, 80, 200, 400, 600, and 800 µg/kg.

7.5.1.3 The standard concentration for compounds in 8260 Mix #4A are 50, 100, 250, 500, 750, and 1000 µg/kg.

7.5.1.4 The standard concentration for compounds in 8260 Mix #5 are 250, 500, 1250, 2500, 3750, and 5000 µg/kg.

7.5.1.5 The standard concentration for compounds in 8260 Mix #6 are 200, 400, 1000, 2000, 3000, and 4000 µg/kg.

7.5.1.6 The standard concentration for compounds in Mix #7 are 100, 200, 500, 1000, 2000 µg/kg.

7.5.2 Composition of Standard Mixtures used in this calibration.

7.5.2.1 Compounds in 8260 Mix #1 at a concentration of 100 µg/ml

- 1,1-dichloroethene
- methylene chloride
- trans-1,2-dichloroethene
- 1,1-dichloroethane
- 2,2-dichloropropane
- cis-1,2-dichloroethene
- bromochloromethane
- chloroform
- 1,1,1-trichloroethane
- carbon tetrachloride
- 1,1-dichloropropene
- benzene
- 1,2-dichloroethane
- trichloroethene
- 1,2-dichloropropane
- dibromomethane
- bromodichloromethane

- cis-1,3-dichloropropene
- toluene
- trans-1,3-dichloropropene
- 1,1,2-trichloroethane
- tetrachloroethene
- 1,3-dichloropropane
- dibromochloromethane
- 1,2-dibromoethane
- chlorobenzene
- 1,1,1,2-tetrachloroethane
- ethylbenzene
- m,p-xylene
- o-xylene
- styrene
- bromoform
- isopropyl benzene
- bromobenzene
- 1,1,2,2-tetrachloroethane
- 1,2,3-trichloropropane
- n-propyl benzene
- 2-chlorotoluene
- 4-chlorotoluene
- 1,2,4-trimethyl benzene
- 1,3,5-trimethyl benzene
- sec-butyl benzene
- 1,2-dichlorobenzene
- 1,3-dichlorobenzene
- 1,4-dichlorobenzene
- n-butylbenzene
- tert-butyl benzene
- p-isopropyl toluene
- 1,2-dibromo-3-chloropropane
- 1,2,4-trichlorobenzene
- hexachlorobutadiene
- naphthalene
- 1,2,3-trichlorobenzene

7.5.2.2 Compounds in 8260 Gases at a concentration of 100 µg/ml

- dichlorodifluoromethane
- chloromethane
- vinyl chloride
- bromomethane
- chloroethane
- trichlorofluoromethane

7.5.2.3 Compounds in 8260 Mix #2 at a concentration of 100 µg/ml

- pentachloroethane
- pentafluorobenzene
- benzyl chloride
- carbon disulfide
- iodomethane
- 1,1,1-trichloro-2,2,2-trifluoroethane
- 1,1,2-trichloro-1,2,2-trifluoroethane
- acetonitrile
- 1-chlorobutane
- 1-chlorohexane
- 3-chloropropene
- n-hexane
- 1,2-diethylbenzene
- methyl-t-butyl ether

7.5.2.4 Compound in 8260 Mix # 2B at a concentration of 100 µg/mL

- vinyl acetate

7.5.2.5 Compounds in 8260 Mix #3A at a concentration of 100 µg/mL, except THF- 400 µg/mL

- acetone
- 4-methyl-2-pentanone
- 2-hexanone
- 2-butanone
- tetrahydrofuran

7.5.2.6 Compounds in 8260 Mix #4A at a concentration of 1000 µg/mL

- methacrylonitrile
- diethylether
- methyl acrylate
- ethylmethacrylate
- methylmethacrylate
- crotonaldehyde
- tert-butyl alcohol

7.5.2.7 Compound in 8260 Mix # 4C (1301B) at a concentration of 100 µg/mL

- 2-chloroethyl vinyl ether

7.5.2.8 Compound in 8260 Mix #5 at a concentration of 5000 µg/ml

- isobutyl alcohol
- 1,4-dioxane
- ethyl cyanide

7.5.2.9 Compounds in 8260 Mix #6 at a concentration of 4000 µg/mL

- cis-1,4-dichloro-2-butene
- trans-1,4-dichloro-2-butene

7.5.2.10 Compounds in 8260 Mix #7 at a concentration of 1000 µg/mL

- acrolein
- acrylonitrile

7.6 Initial Calibration Verification

7.6.1 The initial calibration curve must be verified using a standard from an independent source. The laboratory purchases the initial calibration verification (ICV) standard from a different vendor than the one used for the calibration standards.

7.6.2 The ICV contains the full list of target analytes at the same concentration as the continuing calibration verification (CCV) standard.

7.7 Compounds in 8260 I.S. (Internal standard) at a concentration of 250 µg/ml

- fluorobenzene
- D5-chlorobenzene
- D4-1,4-dichlorobenzene

7.8 Compounds in 8260 S.S. (Surrogate standard) at a concentration of 50 µg/ml

- dibromofluoromethane
- D4-1,2-dichloroethane
- D8-toluene
- 4-bromofluorobenzene

7.9 Compounds in 8260 Spiking Mixture (1001C) at a concentration of 25 µg/mL

- 1,1-dichloroethene
- trichloroethene
- benzene
- toluene
- chlorobenzene

Note: The spiking cocktail is project dependent. This spiking mixture can also be used as a Laboratory Control Sample (LCS) spike. Some projects may require full analyte spike, and in that case, the standard used for the full analyte spike LCS is the ICV. For some programs, the CCV may be used in the place of the LCS.

7.10 Standard Storage

7.10.1 Store the stock standards in Teflon- sealed screw-cap bottles with zero headspace at -10°C to -20°C. Protect the standards from light. Standards for gases usually need to be replaced after one week or as recommended by the manufacturer, unless the acceptability of the standard can be documented. Standards for the non-gases should be monitored and fresh standards prepared if a 20% difference/drift is experienced. These standards need to be replaced after six months or as recommended by the manufacturer, unless the acceptability of the standard can be documented. CEVE and styrene may have to be prepared more frequently.

7.10.2 Store secondary dilution standards in Teflon-sealed screw-cap bottles with minimal headspace at -10°C to -20°C. Protect the standards from light.

The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to their use in preparing the working calibration standards. Prepared from stock solution, they are stored with minimal headspace and replaced after one week. Secondary standards for gases should be replaced after one week unless the acceptability of the standard can be documented.

7.10.3 Working standards must be prepared just prior to analysis unless they are to be purged by an autosampler. When an autosampler is used, the standards may be kept up to 12 hours in purge vessels connected via the autosampler to the purge and trap device. If premixed certified solutions are used store according to manufacturer's documented holding time and storage temperature recommendations.

7.10.4 Purgeable standards are stored in GC/MS VOA Freezer #1, separate from other standards and samples.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All samples must be analyzed within 14 days of collection. **For NYSASP all samples must be analyzed within 10 days of collection.**

8.3 Prior to analysis, all samples must be stored under refrigeration at 2-4.4° C in the reach-in storage unit in the laboratory. After analysis, samples are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Surrogates

9.1.1 Surrogate compounds are added to all samples, QC, and standards prior to analysis. Surrogates are used to assess the efficiency of the analytical system.

9.1.2 Surrogate compounds must meet recovery criteria as shown below (Table 3).

Surrogate Compound	Soil % Recovery Range
Dibromofluoromethane	**33-150%**
D4-1,2-dichloroethane	**43-145%**
D8-toluene	**55-125%**
4-bromofluorobenzene	**46-150%**

9.1.2.1 For the NYSASP, the system monitoring compound (surrogate) recovery criteria are those in the current EPA CLP SOW for Multi-Media, Multi-Concentration Organics, shown in the following table (Table 4).

Surrogate Compound	Soil % Recovery Range
D4-1,2-dichloroethane	**70-121%**
D8-toluene	**84-138%**
bromofluorobenzene	**59-113%**

9.2 Internal Standards

9.2.1 The integrated areas of the quantitation ions of the internal standards are monitored in continuing calibration verification checks, samples, and QC for a change in retention time and response or sensitivity. These should remain reasonably constant over time.

Internal standard retention time and area responses must be assessed in each continuing calibration verification standard by comparison to the corresponding internal standard in the most recent initial calibration mid-point standard. Internal standard responses in samples and QC are compared to the most recent continuing calibration verification.

9.2.2 The area responses of the internal standards must be within 50-200% difference of the area responses compared to.

9.2.3 The retention time for the internal standards must be less than 30 seconds.

9.2.4 If any of these criteria cannot be met, the analytical system must be checked for malfunctions and corrections made. Re-analysis of any affected sample is required.

9.3 Method/Instrument Blanks

9.3.1 Before any samples are analyzed, it must be demonstrated that a laboratory reagent blank is free from contamination that would prevent the determination of any analyte of concern. Sources of background contamination are glassware, purge gas, sorbents, and equipment. Background contamination must be reduced to an acceptable level before proceeding with the next analysis. In general, background from method analytes should be below the reporting limit.

9.3.2 All blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, and continuing calibration verification acceptance criteria.

9.3.3 A method blank is analyzed with each batch of up to 20 samples processed as a group within a 12-hour tune. If more than 20 samples are analyzed in a tune batch, a second method blank is required. Method blanks must be analyzed immediately following a valid continuing calibration verification analysis.

9.3.4 The concentration of the target compounds in the blank must be less than the reporting limit for each target compound except the common lab solvents, methylene chloride and acetone. These must be less than twice the reporting limit.

For NYSASP, the contamination criteria are those presented in the current EPA CLP SOW for Multi-Media, Multi-Concentration Organics.

9.3.5 All samples processed within the same 12-hour tune associated with a method blank that does not meet the blank technical acceptance criteria must be reanalyzed.

9.3.6 Method interferences caused by contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms must be eliminated. The chromatographic system must be inspected for malfunctions, and corrections must be made as required before more samples are analyzed. An instrument blank is analyzed after a high concentration sample in order to eliminate carryover.

9.4 Laboratory Control Sample

9.4.1 A laboratory control sample (LCS, or blank spike, BS, or matrix spike blank for NYSASP) is prepared and analyzed with each tune batch of up to 20 samples.

9.4.2 The solid LCS is prepared at 50 µg/kg with 50 µg/kg of surrogates.

9.4.3 Unless specified by a client or program, a subset of compounds is spiked into the LCS. **The percent recovery criteria for the subset are shown below.**

LCS Spike Compound	Percent Recovery Range 5 gram soil
1,1-Dichloroethene	75-138
Trichloroethene	75-121
Benzene	75-129
Toluene	76-119
Chlorobenzene	78-122

9.4.3.1 Statistical control limits for the remainder of the analytes in the full list LCS are listed in Attachment 2. Gases and other the known "poor" purging compounds are listed below.

9.4.3.2 Poor purging compounds

- gases: bromomethane
  chloromethane
  chloroethane
  vinyl chloride
  dichlorodifluoromethane
  trichlorofluoromethane)
- acetone
- 2-butanone
- carbon disulfide
- crotonaldehyde
- 1,2-dibromo-3-chloropropane
- 1,4-dioxane
- isobutyl alcohol
- 2-hexanone
- 4-methyl-2-pentanone
- vinyl acetate

- 2-chloroethyl vinyl ether

9.4.3.2 When the LCS fails to meet the criteria, the entire batch associated with it must be re-prepared and reanalyzed.

For NYSASP, the matrix spike blank recovery criteria are the same as the matrix spike criteria shown below. When the matrix spike blank fails criteria it must be re-prepared and re-analyzed along with the matrix spikes. Associated samples are not required to be re-processed.

9.5 Matrix Spikes

9.5.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared and analyzed with every SDG.

9.5.2 For the MS/MSD, in addition to spiking internal standard solution and surrogate solution, also add 5.0 µl of 8260 spike solution. For a full LCS requirement, use the ICV standard. The spiking solutions are added by piercing the septum with the syringe needle.

9.5.3 Matrix spikes have the following advisory recovery criteria as shown in Table 6A.

Spike Compound	% Recovery Range
1,1-dichloroethene	**59-172**
trichloroethene	**62-137**
Benzene	**66-142**
toluene	**59-139**
chlorobenzene	**78-122**
All others	**50-150**

9.5.4 Matrix spikes have the following advisory RPD criteria as shown in Table 6B.

Spike Compound	% RPD
1,1-dichloroethene	22
trichloroethene	24
benzene	21
toluene	21
chlorobenzene	21
All others	25

9.5.5 Most spike compounds should meet these criteria. If the criteria are not met in the MS/MSD but are met in the LCS, the results may be reported with the failures attributed to the matrix of the sample. If the LCS does not meet criteria, then all will have to be repeated as discussed above.

9.6 Duplicates

9.6.1 Duplicates, at a frequency of 5%, are required when processing samples submitted to meet the regulatory requirements of North Carolina DENR. This can be satisfied with the MS/MSD.

9.7 Initial Calibration Verification

9.7.1 A second source initial calibration verification (ICV) standard is run after the initial calibration standards have met criteria.

9.7.2 The ICV must be within 20% of its expected value for each target analyte and surrogate or within 40% for the poor purgers and the gases. Sporadic failure of up to three target compounds is allowed but they must not exceed 40% of their expected value. Poor purgers are listed above.

10.0 Calibration and Standardization

10.1 BFB Tuning

10.1.1 The analysis of the instrument performance check solution is performed by injecting 50 ng of BFB (2ul STD ID#7008) into the GC using a 10-µl Hamilton syringe. BFB may be analyzed simultaneously with a continuing calibration verification standard as long as all QC criteria are met.

10.1.2 The peak selection criteria for BFB analysis are as follows (in order of preference):

10.1.2.1 Average one scan prior to the apex of the BFB peak to one scan after the apex, subtracting a single background scan prior to the peak, but no more than 20 scans prior to the elution of BFB. Also, do not subtract part of the BFB peak.

Note: For work performed to comply with the requirements of the NYSSP, the U.S. Army Corps of Engineers (USACE), and the State of West Virginia, only this option is allowed.

10.1.2.2 Choose the apex of the BFB peak only and include background subtraction. *

* Background subtraction is performed to eliminate interference and when performed, the subtracted scan must be no more than 20 scans prior to the elution of the BFB and no scans within the BFB peak may be subtracted.

10.1.2.3 Choose a single scan or a range of scans within the BFB peak and include background subtraction.*

10.1.3 The analysis of the instrument performance check solution must meet the ion abundance criteria given in Table 7.

Table 7: BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 - 40% of M/Z 95
75	30 - 60% of M/Z 95
95	Base Peak; 100% relative abundance
96	5 - 9% of M/Z 95
173	<2% of M/Z 174
174	>50% of M/Z 95
175	5 - 9% of M/Z 174
176	>95% but less than 101% of m/z 174
177	5 - 9% of M/Z 176

10.1.4 Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2) provided that method performance isn't adversely affected.

10.1.5 All criteria must be met according to requirements established by the U.S. EPA shown above. BFB technical acceptance criteria must be met before any standards, samples, or required blanks are analyzed.

GC/MS tuning and Mass Calibration forms must be printed and attached to the instrument runlog page for each tune. The relative abundance for each ion is calculated to two decimal places.

10.1.6 If BFB technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source, clean the quadrupole rods, or take other corrective action to achieve the technical acceptance criteria.

10.2 Initial Calibration

10.2.1 Prior to the analysis of samples and required blanks, and after the instrument performance check solution (BFB) criteria have been met, each GC/MS system must be calibrated at five concentrations to demonstrate instrument sensitivity and the linearity of responses for the purgeable target compounds.

10.2.2 Prepare standards according to the Initial Calibration Standard Preparation Table 2 in Section 7.5. All initial calibration standards must be analyzed at the concentration levels and frequency described in this SOP on a GC/MS system meeting the BFB technical acceptance criteria. The analysis of the five (or six) calibration standards determines the linearity of the five-point initial calibration curve.

10.2.3 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard. Relative response factors (RRF) are calculated for each compound.

10.2.4 Initial calibration technical acceptance criteria must be met before any samples or required blanks are analyzed.

For analyses following the NYSASP, the initial calibration requirements, after meeting the instrument performance check (tune) requirements of this SOP, are those of the current EPA CLP SOW for Multi-Media, Multi-Concentration Organics (for 1-5 g analyses).

10.2.5 Minimum relative response factors for the System Performance Check Compounds (SPCCs) are listed in Table 8.

Table 8: Relative Response Factor Criteria for SPCCs

Volatile Compound	Minimum RRF
Chloromethane	0.10
1,1-dichloroethane	0.10
chlorobenzene	0.30
bromoform	0.10
1,1,2,2-tetrachloroethane	0.30

10.2.6 The %RSD for each target analyte should be less than 15%, but the following compounds have maximum %RSD criteria of 30%: These Calibration Check Compounds (CCC) include:

- vinyl chloride
- 1,1-dichloroethene
- chloroform
- 1,2-dichloropropane
- toluene
- ethylbenzene

10.2.6.1 While the remaining target compounds do not have defined % RSD criteria, a warning limit of 50% RSD and an action limit of 90% RSD have been inserted as default values into the data reduction software program. This is based strictly on established U.S. EPA data validation guidelines where values greater than 90% RSD result in rejection of data.

10.2.6.2 If the % RSD is 15% or less, the average relative response factor may be used for quantitation. If the % RSD is greater than 15% then an alternate method for quantitation, such as least squares regression or a non-linear calibration method, may be used. When one of these options is used, the correlation coefficient of the equation must be 0.99 or greater for a valid calibration. If a quadratic equation is used, six levels of standards must be employed. If a third degree polynomial is used, seven levels are required. These alternate methods of quantitation are available in the ThruPut system.

For samples submitted to meet the regulatory requirements of the State of South Carolina, the option of using a quadratic fit to demonstrate linearity is not allowed. However, the use of a linear regression analysis for each target analyte is allowed.

10.2.6.3 Because of the large number of target analytes, some of them may exceed the 15% criteria. When this occurs, certain steps may be performed. These corrective actions also pertain to those instances where the 90% RSD action limits have been exceeded by the non-criteria compounds.

- Check the instrument operating conditions and perform maintenance as necessary. It may be necessary to clean the ion source, perform column maintenance, change the column, service the Archon autosampler, or the purge and trap concentrator, or take other corrective action to achieve the technical acceptance criteria.

- Compare responses for the analyte in each of the standard levels to verify that a single standard analysis is not producing the outliers. If so, reanalyze that standard and recalculate the %RSD.

- The calibration range may be narrowed to determine if linearity can be achieved. This may cause more dilution re-analyses or even change the reporting limit if the lower standard is eliminated. For this method, the method quantitation limit is defined by the lowest standard.

10.2.7 The initial calibration may still be acceptable when some analytes exceed the 15% RSD criteria, if the following conditions are met:

- The mean of <u>all</u> %RSD values for the analytes is less than or equal to 15%.

- All analytes in calibration the standard must be included in the calculation.

10.2.7.1 A summary of the initial calibration data and/or a list of the analytes not meeting the 15% RSD criteria and the actual %RSD for each of these analytes must be included as a deliverable to our client. If the conditions in 10.2.7 are met, then the average relative response factor may be used to determine the concentration of analytes in samples.

10.2.7.2 For samples submitted to meet the regulatory requirements of the State of South Carolina, the option of demonstrating that the mean %RSD of all target analytes in the standard mixture is under 15% is not an allowed option. Because the State of South Carolina does not allow the grand mean, the following acceptance criteria for the initial calibration apply to samples received for compliance with the DHEC.

Of the remaining non-CCC analytes the %RSD should be less than 15%, but the following compounds have maximum %RSD criteria of 30%. These compounds are the gaseous analytes and those characterized with having poor purging efficiencies or poor responses.

- dichlorodifluoromethane
- chloromethane
- vinyl chloride
- bromomethane
- chloroethane
- trichlorofluoromethane
- acrolein
- iodomethane
- carbon disulfide
- acetone
- acetonitrile
- acrylonitrile
- tert-butyl alcohol
- vinyl acetate
- 2-butanone
- propionitrile
- methyl acrylate
- methacrylonitrile
- isobutyl alcohol

- crotonaldehyde
- 1,4-dioxane
- methylmethacrylate
- 2-chloroethyl vinyl ether
- 4-methyl-2pentanone
- ethylmethacrylate
- 2-hexanone
- benzyl chloride
- 1,2-dibromo-3-chloropropane

The exceptions to the 15% mean %RSD criteria discussed above in Section 10.2.6 are also allowed by the State of South Carolina.

10.2.8 The initial calibration verification must be analyzed after each initial calibration and must meet the acceptance criteria. The ICV establishes the validity of the curve. If the ICV fails, then a new initial calibration curve must be generated.

10.3 Continuing Calibration Verification

10.3.1 Before the analysis of samples and blanks, but after BFB and initial calibration acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing a continuing calibration verification standard. This standard contains all purgeable target analytes and surrogate compounds. It is used to ensure that the instrument meets the sensitivity and linearity requirements of the method throughout the analytical sequence.

10.3.2 A check of the calibration curve must be performed once every 12 hours, beginning with the injection of BFB. A percent difference of the response for each compound compared to the mean relative response factor from the initial calibration is calculated when performing the average response factor model.

10.3.3 The calculated percent difference must be less than or equal to 20% for the CCCs listed above in Section 10.2.6. Minimum response factor criteria for the continuing calibration verification standard are also shown above in Table 8.

10.3.3.1 If a regression fit model was used for analytes in the initial calibration, the continuing calibration verification is performed using percent drift (difference) for the CCCs.

10.3.4 As indicated for the initial calibration acceptance criteria, for the continuing calibration verification, the remaining target analytes (non-CCC compounds) do not have defined % difference criteria. We have established a warning limit of 50%D and an action limit of 90%D. These values have been inserted as defaults into the data reduction software program. This is based strictly on established U.S. EPA data validation guidelines where values greater than 90% RSD results in rejection of data.

10.3.4.1 For samples submitted to meet the regulatory requirements of the State of South Carolina, the non-CCC target analytes should be less than 20%. Compounds listed in 10.2.7.2 have a warning limit of 40% D and an action limit of 50% D.

10.3.5 If continuing calibration verification acceptance criteria cannot be met after inspection and normal maintenance, a new initial calibration will have to be performed.

Note: Method 8260B indicates that if the CCCs are not required analytes, then all required analytes, must meet the 20% difference/drift criterion. Our typical analysis includes all of the CCCs. Additionally, some programs may require all compounds to meet a % difference criteria. In these situations, if the average of the response for all analytes is within 20%, then the calibration has been verified. Requirements similar to those in 10.2.6 must be met.

10.3.6 For analyses following the NYSASP, the continuing calibration requirements, after meeting the instrument performance check (tune) requirements of this SOP, are those of the current EPA CLP SOW for Multi-Media, Multi-Concentration Organics (for 1-5g analyses).

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. All injections must be recorded on the instrument runlog (Attachment 4) along with the date, time (use a 24 hour clock), the volume injected, operator ID, and any comments relevant to the injection.

All samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration verification, and instrument blank criteria.

11.1 Instrument Software Conventions

11.1.1 Quantitation method: Average of the whole

11.1.2 File naming convention: XX012345YZZ

where: **XX =** Analytical prefix
where:
Calibration tune file = BF,BG,...
Instrument blank = CB,CC,...
Calibration standard = CS,CT,...
Initial sample analysis = CN
Sample reanalysis = CR0,C2R,C3R

Y = Shift indicator = A,B, or C
ZZ = Instrument number
12345 = Last five digits of lab ID

11.2 Analytical Sequence

11.2.1 Order of analysis for the instrument calibration

- BFB (tune)
- initial calibration
- initial calibration verification

11.2.2 Order of analysis for the twelve-hour tune

- BFB
- continuing calibration verification
- instrument blank
- laboratory control sample-LCS
- samples

11.2.3 In some cases, if tune time remains after the initial calibration standards have been run, samples may be analyzed as long as they are preceded by a valid instrument blank.

11.2.4 All samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration verification, and instrument blank criteria.

11.3 Preparations

11.3.1 Standards

11.3.1.1 The analysis of the instrument performance check solution is performed by injecting 50 ng of BFB (2ul Standard ID#7008) into the GC using a 10-µl Hamilton syringe.

11.3.1.2 Calibration standards are prepared by spiking the appropriate volume of each standard solution into 5 mL of sparged DI water contained in a 5 mL syringe. This is then added to a 40 mL vial containing a magnetic stirring bar and 1g of sodium bisulfate. Each vial is immediately capped with a PTFE-lined, septum-sealed cap and loaded into the Archon autosampler.

Initiate the Archon autosampler which will provide stirring, the addition of 5 mL of water containing internal standards, heating at 40°C, and purging for 11 minutes. The system will then transfer the constituents in the headspace to the Tekmar 3000 purge and trap concentrator and will then desorb all target analytes for 4 minutes before analysis. The analysis of the five (or six) calibration standards determines the linearity of the five-point initial calibration curve.

11.3.2 Instrument Blank and Method Blank

11.3.2.1 An instrument blank is prepared by filling a 40 mL VOA vial, containing a stir bar and 1g of sodium bisulfate, with 5 mL of purged DI water and sealing with a screw-top, PFTE-faced, septum-sealed cap. This is placed into the Archon autosampler where DI water, internal standards, and surrogates are added automatically to the blank. It is analyzed by a closed system heated purge and trap analysis.

11.3.2.2 A Method Blank is similar to an Instrument Blank in composition but it is prepared at the same time samples are prepared and is stored in the refrigerator. If samples are received from the field already in vials with the sodium bisulfate preservative solution, only an Instrument Blank is required.

11.3.3 Laboratory Control Sample

11.3.3.1 A laboratory control sample (LCS) is prepared by filling a 40 mL VOA vial, containing a stir bar and 1g of sodium bisulfate, with 5 mL of purged DI water. To this 10 µl of the spiking standard is added before sealing with a screw-top, PFTE-faced, septum-sealed cap. This is placed into the Archon autosampler where DI water, internal standards, and surrogates are added automatically. It is analyzed by a closed system heated purge and trap analysis.

11.3.3.2 For certain projects and programs, a full list spike is required.

11.3.4 Samples

11.3.4.1 Solid samples are prepared by Method 5035. For details see Sample Preparation Procedure –238: "Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap." This results in 5g and 1g samples being stored in separate sealed 40 mL VOA vials containing sample, a stirring bar, and a sodium bisulfate aqueous solution (0.2g sodium bisulfate per g of sample).

11.3.4.2 The choice of whether a 5g or 1g sample is analyzed is generally based on a screen analysis.

11.3.4.3 Samples are stored in a rack located in the volatile GC/MS laboratory refrigerator at 2°C to 4.4°C. Samples are allowed to come to room temperature and then loaded into the Archon autosampler carousel shaking each vial gently so that the contents move freely and the stirring bar will be able to spin.

11.4 Matrix Spikes

11.4.1 For sample spikes, in addition to spiking internal standard solution and surrogate solution, also add 10.0 µl of 8260B spike solution.

11.4.2 For certain projects, a full target list matrix spikes are required.

11.4 Analysis

11.4.1 When the Archon autosampler is initiated, the system will add 5 mL of purged DI water, containing internal standards and surrogates, by piercing the septum.

11.4.2 Prior to purging, the stirring bar is turned on and the sample is heated to 40°C. The sample is purged for 11 minutes, while stirring.

11.4.3 The same needle that is used to add the DI water is the source for the inert gas used for purging. The needle also contains slots above the sample/water level which provide a path for the headspace to be directed to the Tekmar 3000 purge and trap concentrator. This contains the trap that is then thermally desorbed into the GC/MS instrument.

11.4.4 After purging, the Purge and Trap Concentrator apparatus will desorb onto the GC column by elevating the trap temperature to 260°C and back-flushing the trap with helium for 4 minutes at 20 to 60 mL/minute.

11.4.5 After desorbing, the trap is reconditioned by baking at 260°C for at least 7 minutes. When the trap has finished baking and is cool, it is ready for the next sample to be purged.

11.4.6 In each analytical run, all analytes must fall below the maximum calibration range established by the highest standard in the initial calibration. If an analyte is present at a concentration higher than the highest initial calibration standard, it must be reanalyzed at a lesser amount or dilution. A valid dilution is one in which the compound in question falls above the mid-point calibration standard concentration. The dilution is considered valid if the analyte concentration is above 50 µg/kg.

11.5 Identification

11.5.1 Target compounds are identified in the samples by analyzing standards under the same conditions used for samples. The resulting mass spectra are compared to established library spectra and GC retention times to retention times from the latest continuing calibration standard. The mass spectrum of the sample compound and a laboratory library-generated spectrum must match according to the following criteria:

11.5.1.1 All ions present in the library mass spectrum at a relative intensity >10% must be present in the sample spectrum.

11.5.1.2 The relative intensities of ions specified above must agree within ±20% between the library and sample spectra.

11.5.1.3 Ions >10% in the sample spectrum but not present in the library spectrum must be considered and accounted for.

11.5.2 If a compound analyzed by GC/MS techniques cannot be verified by all of the criteria listed above, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the laboratory will report that identification.

11.5.3 Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology (NIST) Mass Spectral Library.

11.6 Quantitation

11.6.1 The mean relative response factor (RRF) from the initial calibration standard is used to calculate the concentration in the sample. For NYSASP, the RRF from the continuing calibration standard is used to calculate concentrations.

Note: Alternatively, the calibration curve(s) generated from the initial calibration may be used for the determination of analyte(s) concentration(s). This option is discussed above.

11.6.2 All samples require a search of all extraneous peaks >10% of the height of the nearest internal standard, up to 10 searches, i.e. 10 most intense extraneous peaks. The number of searches may be more, depending on client requirements.

11.6.3 In each analytical run, all analytes must fall below the method's maximum analytical range, i.e. the highest calibration standard.

11.6.3.1 If an analyte is present at a concentration higher than the maximum analytical range in a 5g analysis, the 1g sample must be analyzed.

11.6.3.2 If an analyte is still present at a concentration higher than the highest standard in the 1g analysis, a medium concentration procedure (methanol extraction) must be followed.

11.6.4 When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by the analysis of an instrument blank or the following sample must be monitored for contamination and interference from carryover. If the blank or sample is not free from interferences, the system must be decontaminated. Sample analysis may not resume until a blank or sample has been analyzed which is free from interferences. Being free from interferences means that whatever compound was present above the initial calibration range in a sample, cannot be present in an instrument blank or the sample analyzed immediately following, at a level above the reporting limit for that compound.

11.6.5 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG narrative.

11.6.6 Non-target compounds are quantified by comparing the MS response from the reconstructed ion chromatogram (RIC) for the non-target compound peaks to the MS response for a peak produced by the nearest internal standard compound. A response factor of 1 is assumed.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{\textit{Amount found}}{\textit{Amount spiked}}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\,\mathrm{R} = \frac{\textit{Amount in spiked sample - Amount in unspiked (native) sample}}{\textit{Amount spiked}}\ x\ 100$$

12.4 Calculation of % RSD

$$\%\mathrm{RSD} = \left(\frac{\textit{Standard deviation}}{\overline{\mathrm{X}}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{(Value\ 1 + Value\ 2)/2} x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}}\ x\ 100$$

12.7 Relative Response Factor

$$RRF = \frac{Ax\ x\ C(is)}{A(is)\ x\ Cx}$$

where: Ax = Area of the characteristic ion (EICP) for the compound to be measured

A(is) = Area of the characteristic ion (EICP) for the specific internal standard

C(is) = Concentration of the internal standard (in µg/l)

Cx = Concentration of the compound to be measured

12.8 Concentration

12.8.1 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard.

12.8.2 Concentration of soil samples (dry weight basis) by GC/MS

$$ug / kg = \frac{(Ax)(Is)(Vt)(Df)\cancel{(2.0)}}{(Ais)(RRF)(Vi)(Ws)(D)}$$

where: Ax, Ais, ~~Is, V~~t, Vi, RRF, and Df are the same as given for water
~~2.0 = GPC factor (if used)~~
Ws = weight of sample extracted, in grams
D (dry weight)= $\frac{100 - \%\ moisture}{100}$

12.8.3 Tentatively Identified Compound (TIC) Estimation

$$TIC\ Amount = \frac{(Area\ TIC)\ x\ Amount(Std)}{Area(IS)\ x\ 1(RF)}$$

where: Area(TIC) = area response from RIC for non-target compound
Amount(Std) = amount of internal standard added to the sample, in µg/L.
Area(IS) = area response of the nearest internal standard in the reconstructed ion chromatogram
1(RF) = assumed response factor of 1

12.9 Calculating Dilutions

If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. A 10x dilution is performed using 1 mL sample plus 9 mL diluent for a total volume of 10 mL. It should be recorded on the run log as "10x (1 mL in 10 mL)."

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits (Attachment **3**) and precision and accuracy for single analyst (Attachment **5**). The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. U.S. EPA SW846 3rd Edition, Update 3, 12/96, Methods 8260B and 5035

16.2 New York State Analytical Services Protocol (NYSASP), June 2000

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 QCSOP: Proper Documentation Procedures

16.5 QCSOP: Numerical Data Reduction

16.6 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.7 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.8 NELAC Standards, July 1, 1999, plus revisions

16.9 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.10 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.11 CompuChem Quality Manual, Revision 2, 10/26/01, plus revisions

16.12 Sample Control SOP 4.1, "Receiving Samples"

16.13 Sample Control SOP 4.6, "Storing Samples"

16.14 Sample Preparation Procedure –238: "Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap."

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 – Target Compound List

17.2 Attachment 2 – **Statistical Control Limits**

17.3 Attachment 3 - Method Detection Limits

17.4 Attachment 4 – Instrument Runlog

17.5 Attachment 5 - Single Analyst Capability Study

Attachment 1

Table 1: Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
dichlorodifluoromethane	1	85	87
chloromethane	1	50	52
vinyl chloride	1	62	64
bromomethane	1	94	96
chloroethane	1	64	66
trichlorofluoromethane	1	101	103
1,1-dichloroethene	1	96	61, 98
methylene chloride	1	84	49, 86
trans-1,2-dichloroethene	1	96	61, 98
1,1-dichloroethane	1	63	65, 83
2,2-dichloropropane	1	77	97
cis-1,2-dichloroethene	1	96	61, 98
bromochloromethane	1	128	49, 130
chloroform	1	83	85
1,1,1-trichloroethane	1	97	99, 61
carbon tetrachloride	1	117	119, 121
1,1-dichloropropene	1	75	110, 77
benzene	1	78	77, 51
1,2-dichloroethane	1	62	98
trichloroethene	1	130	95, 97
1,2-dichloropropane	1	63	112
dibromomethane	1	174	93, 95
bromodichloromethane	1	83	85, 127
2-chloroethyl vinyl ether	1	63	65, 106
cis-1,3-dichloropropene	1	75	77
acrolein	1	56	55, 58
iodomethane	1	142	127, 141
1,1,1-trichloro-2,2,2,-trifluoroethane	1	117	151, 153
1,1,2-trichloro-1,2,2,-trifluoroethane	1	85	101, 151
carbon disulfide	1	76	78
acetone	1	43	58
3-chloropropene	1	76	41, 78
acetonitrile	1	41	40, 39

Attachment 1 (continued)

Table 1 (continued): Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
Acrylonitrile	1	53	52, 51
methyl-tert-butyl ether	1	73	41, 43
vinyl acetate	1	43	86
2-butanone	1	72	43, 57
Propionitrile	1	54	55, 52
Methacrylonitrile	1	41	39, 67
1-chlorobutane	1	56	49
1,4-dioxane	1	88	58
Methylmethacrylate	1	69	100, 41
Surrogate #1: Dibromofluoromethane	1	113	111, 192
Surrogate #2: d4-1,2-dichloroethane	1	65	102, 67
4-methyl-2-pentanone	2	43	85, 100
Toluene	2	92	91
trans-1,3-dichloropropene	2	75	77
1,1,2-trichloroethane	2	97	83, 85
Ethylmethacrylate	2	69	41, 99
Tetrachloroethene	2	164	168, 129
1,3-dichloropropane	2	76	78
2-hexanone	2	43	58, 57
Dibromochloromethane	2	129	127, 48
1,2-dibromoethane	2	107	109, 188
Chlorobenzene	2	112	114, 77
1,1,1,2-tetrachloroethane	2	131	119, 133
Ethylbenzene	2	106	91
m,p-xylene	2	106	91
o-xylene	2	106	91
Styrene	2	104	91, 78
Bromoform	2	173	175, 254
isopropyl benzene	2	105	120
Bromobenzene	2	156	77,158
1,1,2,2-tetrachloroethane	2	83	85, 131

Attachment 1 (continued)

Table 1 (continued): Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
1,2,3-trichloropropane	2	110	75, 112
trans-1,4-dichloro-2-butene	2	53	88, 75
Surrogate #3: d8-toluene	2	98	70, 100
n-propyl benzene	3	91	120
2-chlorotoluene	3	126	91
4-chlorotoluene	3	91	126
1,2,4-trimethyl benzene	3	105	120
1,3,5-trimethyl benzene	3	105	120
Pentachloroethane	3	167	130, 165
sec-butyl benzene	3	105	134
1,2-dichlorobenzene	3	146	111, 148
1,3-dichlorobenzene	3	146	111, 148
1,4-dichlorobenzene	3	146	111, 148
n-butyl benzene	3	91	92, 134
tert-butyl benzene	3	119	91, 134
p-isopropyl toluene	3	119	134, 91
1,2-dibromo-3-chloropropane	3	75	155, 157
1,2,4-trichlorobenzene	3	180	182, 145
Hexachlorobutadiene	3	225	223, 227
Naphthalene	3	128	64, 51
1,2,3-trichlorobenzene	3	180	182, 145
Surrogate #4: 4-bromofluorobenzene	3	95	174, 176

[1] Based on laboratory tests, 2-chloroethyl vinyl ether is not analyzable from the sodium bisulfate solution associated with Method 5035.

Internal Standard #1: fluorobenzene
Internal Standard #2: d5-chlorobenzene
Internal Standard #3: d4-1,4-dichlorobenzene

Attachment 2

Table 2: Statistical Control Limits for the LCS

Compound	Percent Recovery Range 5 gram soil
Dichlorodifluoromethane	50-150
Chloromethane	50-142
vinyl chloride	50-137
Bromomethane	56-139
Chloroethane	66-126
Trichlorofluoromethane	50-150
Diethyl ether	50-149
Acrolein	50-150
1,1-dichloroethene*	75-138
Iodomethane	50-150
1,1,1-trichloro-2,2,2,-trifluoroethane	58-148
carbon disulfide	50-150
1,1,2-trichloro-1,2,2,-trifluoroethane	50-150
Acetone	50-150
3-chloropropene	50-150
Acetonitrile	68-126
Methyl acetate	50-150
methylene chloride	50-150
trans-1,2-dichloroethene	69-135
Acrylonitrile	50-136
methyl-tert-butyl ether	55-150
Tert butyl alcohol	60-148
n-hexane	50-143
1,1-dichloroethane	71-135
Chloroprene	50-150
vinyl acetate	50-150
Isopropyl ether	81-114
2,2-dichloropropane	64-141
cis-1,2-dichloroethene	79-124
2-butanone	50-150
Propionitrile	50-150
Bromochloromethane	73-126
Methyl acrylate	50-133
Methacrylonitrile	51-127
Tetrahydrofuran	50-150

Attachment 2 (continued)

Table 2: Statistical Control Limits for the LCS

Compound	Percent Recovery Range 5 gram soil
Chloroform	74-131
1,1,1-trichloroethane	73-130
Cyclohexane	50-150
1-chlorobutane	75-124
carbon tetrachloride	72-130
1,1-dichloropropene	72-132
Pentafluorobenzene	81-117
Benzene*	75-129
1,2-dichloroethane	60-150
Isobutyl alcohol	50-150
Crotonaldehyde	50-141
Trichloroethene*	75-121
Methylcyclohexane	50-150
1,2-dichloropropane	74-131
Dibromomethane	69-135
1,4-dioxane	50-150
methylmethacrylate	60-127
Bromodichloromethane	75-133
2-chloroethyl vinyl ether	50-150
cis-1,3-dichloropropene	50-150
4-methyl-2-pentanone	50-145
Toluene*	76-119
trans-1,3-dichloropropene	77-126
1,1,2-trichloroethane	75-127
ethylmethacrylate	67-124
Tetrachloroethene	61-124
1,3-dichloropropane	75-130
2-hexanone	50-150
dibromochloromethane	79-122
1,2-dibromoethane	69-131
Chlorobenzene*	78-122
1-chlorohexane	80-113
1,1,1,2-tetrachloroethane	82-120
ethylbenzene	80-125
m,p-xylene	78-130

Attachment 2 (continued)

Table 2: Statistical Control Limits for the LCS

Compounds	Percent Recovery Range 5 gram soil
o-xylene	76-130
Styrene	76-135
Bromoform	76-126
isopropyl benzene	70-140
cis-1,4-dichloro-2-butene	50-150
Bromobenzene	67-140
1,2,3-trichloropropane	58-137
1,1,2,2-tetrachloroethane	67-140
trans-1,4-dichloro-2-butene	50-150
n-propyl benzene	71-136
2-chlorotoluene	78-124
4-chlorotoluene	74-131
1,3,5-trimethyl benzene	72-129
Pentachloroethane	64-150
tert-butyl benzene	67-140
1,2,4-trimethyl benzene	70-133
sec-butyl benzene	67-141
1,3-dichlorobenzene	81-125
1,4-dichlorobenzene	78-123
p-isopropyl toluene	74-132
Benzyl chloride	63-124
1,2-dichlorobenzene	81-123
n-butyl benzene	63-137
1,2-diethylbenzene	75-117
1,2-dibromo-3-chloropropane	50-145
1,2,4-trichlorobenzene	66-131
hexachlorobutadiene	68-129
naphthalene	50-139
1,2,3-trichlorobenzene	63-129
Xylene (total)	75-134

Attachment 3

Method Detection Limit Study

Study date: April 17, 2000			GCMS Volatile SW846 8260B/5035 Soil, 5 gm Purge												
Instrument: F50055															
Compound Name	Rep# 1	Rep# 2	Rep# 3	Rep# 4	Rep# 5	Rep# 6	Rep# 7	Rep# 8	Rep# 9	Rep# 10	Mean	Amt.	S.Dev.	MDL	Report Limit
	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg
Dichlorodifluoromethane	0.15	0.36	0.32	0.29	0.29	0.28	0.27	0.24	0.25	0.21	0.27	2.5	0.058	0.16	10
Chloromethane	0.63	0.93	0.90	0.83	0.85	0.80	0.78	0.77	0.77	0.64	0.79	2.5	0.098	0.28	10
Vinyl Chloride	0.58	1.18	1.11	1.06	1.01	0.97	0.97	0.84	0.89	0.79	0.94	2.5	0.174	0.49	10
Bromomethane	1.55	2.16	2.07	2.04	2.06	1.99	1.97	1.88	1.94	1.81	1.95	2.5	0.172	0.48	10
Chloroethane	1.36	2.13	2.07	2.05	2.02	1.98	1.97	1.86	1.83	1.76	1.90	2.5	0.223	0.63	10
Trichlorofluoromethane	0.88	1.92	1.74	1.69	1.65	1.60	1.49	1.42	1.39	1.27	1.51	2.5	0.290	0.82	10
Diethylether	11.5	13.3	10.6	12.2	13.1	13.0	12.0	12.9	11.2	10.6	12.0	12.5	1.034	2.92	50
Acrolein	16.3	23.5	14.4	18.2	18.8	22.2	14.7	19.4	12.5	11.0	17.1	25.0	4.073	11.49	100
1,1-Dichloroethene	1.20	2.06	1.91	1.89	1.81	1.81	1.71	1.57	1.59	1.47	1.70	2.5	0.251	0.71	10
Iodomethane	1.58	2.30	2.27	2.19	2.11	2.09	2.07	1.95	2.01	1.92	2.05	2.5	0.207	0.58	10
1,1,1-Trichloro-2,2,2-trifluoroethane	0.87	1.86	1.77	1.73	1.67	1.63	1.50	1.37	1.38	1.24	1.50	2.5	0.298	0.84	10
Carbon disulfide	1.13	1.96	1.79	1.67	1.70	1.53	1.59	1.41	1.40	1.26	1.54	2.5	0.251	0.71	10
1,1,2-trichloro-1,2,2-trifluoroethane	0.89	1.90	1.84	1.73	1.70	1.63	1.53	1.41	1.42	1.26	1.53	2.5	0.302	0.85	10
Acetone	5.26	4.71	3.10	4.23	5.14	7.73	4.32	*	3.26	2.92	4.52	2.5	1.482	4.29	10
3-Chloropropene	1.25	1.86	1.74	1.59	1.57	1.58	1.46	1.40	1.44	1.30	1.52	2.5	0.189	0.53	10
Acetonitrile	1.46	1.99	1.81	1.84	1.82	1.99	1.65	1.74	1.64	1.41	1.74	2.5	0.198	0.56	10
Methylene Chloride	2.47	2.71	2.74	2.86	2.82	2.89	2.86	3.10	2.91	2.88	2.82	2.5	0.163	0.46	10
trans-1,2-Dichloroethene	1.53	2.28	2.19	2.10	2.09	2.00	1.96	1.88	1.92	1.81	1.98	2.5	0.212	0.60	10
Acrylonitrile	21.5	25.7	14.4	21.6	25.7	25.9	18.1	26.3	16.4	15.1	21.1	25.0	4.771	13.46	100
Methyl-tert-butyl-ether	2.06	2.34	1.72	2.10	2.21	2.23	1.97	2.15	1.81	1.71	2.03	2.5	0.221	0.62	10
Tert-butyl-alcohol	8.37	11.50	4.08	8.49	12.34	9.77	7.34	13.20	6.47	3.88	8.54	12.5	3.228	9.11	50
n-Hexane	0.92	1.97	1.87	1.84	1.76	1.69	1.64	1.48	1.53	1.38	1.61	2.5	0.304	0.86	10
1,1-Dichloroethane	1.82	2.39	2.28	2.25	2.24	2.18	2.17	2.08	2.13	2.04	2.16	2.5	0.156	0.44	10
Vinyl acetate	0.84	1.02	1.36	1.53	1.54	0.27	0.74	1.16	0.69	0.89	1.00	2.5	0.403	1.14	10
Isopropyl ether	1.61	1.95	1.77	1.80	1.80	1.76	1.76	1.67	1.66	1.63	1.74	2.5	0.102	0.29	10
2,2-Dichloropropane	1.00	1.60	1.38	1.35	1.29	1.22	1.17	1.12	1.08	0.95	1.22	2.5	0.196	0.55	10
cis-1,2-Dichloroethene	1.43	1.81	1.67	1.72	1.62	1.61	1.62	1.53	1.55	1.45	1.60	2.5	0.117	0.33	10
2-Butanone	1.50	1.60	0.50	1.25	1.66	1.62	1.12	2.57	0.71	0.57	1.31	2.5	0.626	1.77	10
Propionitrile	7.39	8.56	4.78	7.01	8.32	8.69	6.02	8.39	5.07	4.66	6.89	12.5	1.636	4.61	50
Bromochloromethane	1.73	2.02	1.72	1.86	1.83	1.87	1.76	1.97	1.73	1.64	1.81	2.5	0.119	0.34	10
Methyl acrylate	8.12	9.51	5.35	7.99	9.25	9.43	6.65	9.34	5.82	5.37	7.68	12.5	1.739	4.90	50
Methacrylonitrile	7.99	9.41	5.49	7.79	9.17	9.28	6.78	9.19	5.96	5.55	7.66	12.5	1.608	4.54	50
Tetrahydrofuran	6.24	7.45	3.68	5.79	7.49	7.55	4.97	7.55	4.14	3.65	5.85	10.0	1.648	4.65	40
Chloroform	1.57	1.97	1.81	1.83	1.79	1.77	1.72	1.67	1.71	1.62	1.75	2.5	0.115	0.32	10
1,1,1-Trichloroethane	1.10	1.78	1.70	1.65	1.56	1.53	1.49	1.39	1.41	1.31	1.49	2.5	0.200	0.57	10
1-Chlorobutane	0.96	1.67	1.49	1.49	1.47	1.44	1.37	1.29	1.31	1.18	1.37	2.5	0.197	0.55	10
Carbon tetrachloride	0.96	1.68	1.64	1.57	1.49	1.40	1.38	1.30	1.27	1.19	1.39	2.5	0.220	0.62	10
1,1-Dichloropropene	0.97	1.65	1.54	1.47	1.44	1.43	1.37	1.28	1.28	1.18	1.36	2.5	0.194	0.55	10
Pentafluorobenzene	1.21	2.07	1.94	1.98	1.85	1.82	1.76	1.66	1.65	1.55	1.75	2.5	0.249	0.70	10
Benzene	1.41	1.81	1.66	1.67	1.66	1.61	1.60	1.61	1.57	1.49	1.61	2.5	0.108	0.30	10
1,2-Dichloroethane	1.68	1.90	1.57	1.72	1.83	1.79	1.68	1.79	1.59	1.54	1.71	2.5	0.119	0.34	10
Isobutyl alcohol	67.1	79.1	33.5	63.2	89.2	51.8	50.9	101.1	44.1	32.7	61.3	125.0	23.09	65.14	500
Crotonaldehyde	3.72	9.70	1.98	3.91	5.18	3.94	3.12	5.80	2.40	2.22	4.20	12.5	2.295	6.47	50
Trichloroethene	1.38	1.91	1.76	1.77	1.73	1.83	1.67	1.56	1.60	1.49	1.67	2.5	0.163	0.46	10
1,2-Dichloropropane	1.52	1.82	1.64	1.65	1.65	1.63	1.60	1.58	1.55	1.47	1.61	2.5	0.095	0.27	10
Dibromomethane	1.70	1.91	1.51	1.75	1.79	1.80	1.62	1.74	1.59	1.50	1.69	2.5	0.133	0.38	10
1,4-Dioxane	57.1	76.9	35.7	58.5	81.6	76.4	51.8	85.0	46.9	38.1	60.8	125.0	18.12	51.11	500
Methylmethacrylate	1.51	1.78	1.07	1.54	1.78	1.77	1.33	1.78	1.17	1.09	1.48	2.5	0.298	0.84	10
Bromodichloromethane	1.50	1.78	1.56	1.63	1.60	1.57	1.50	1.51	1.48	1.40	1.55	2.5	0.103	0.29	10

Attachment 3(continued)

Method Detection Limit Study

Study date: April 17, 2000			GCMS Volatile SW846 8260B/5035 Soil, 5 gm Purge												
Instrument: F50055															
Compound Name	Rep# 1	Rep# 2	Rep# 3	Rep# 4	Rep# 5	Rep# 6	Rep# 7	Rep# 8	Rep# 9	Rep# 10	Mean	Amt.	S.Dev.	MDL	Report Limit
	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg	ug/Kg
2-Chloroethyl vinyl ether	0.33	0.33	0.07	0.28	0.45	0.37	0.23	0.55	0.27	0.13	0.30	2.5	0.141	0.40	10
cis-1,3-Dichloropropene	1.32	1.58	1.31	1.36	1.35	1.35	1.25	1.27	1.20	1.12	1.31	2.5	0.121	0.34	10
4-Methyl-2-pentanone	1.79	2.02	1.08	1.63	1.94	1.69	1.34	2.36	1.22	1.03	1.61	2.5	0.437	1.23	10
Toluene	1.67	2.26	*	2.08	1.96	1.94	1.91	2.23	1.92	1.75	1.97	2.5	0.197	0.57	10
trans-1,3-Dichloropropene	1.27	1.41	1.08	1.28	1.25	1.23	1.12	1.20	1.06	1.01	1.19	2.5	0.122	0.34	10
1,1,2-Trichloroethane	1.79	1.95	1.48	1.79	1.85	1.91	1.69	1.91	1.61	1.49	1.75	2.5	0.173	0.49	10
Ethylmethacrylate	8.56	10.28	6.58	8.95	9.74	8.93	7.80	9.81	7.27	6.69	8.46	12.5	1.323	3.73	50
Tetrachloroethene	1.39	2.15	1.99	1.98	2.02	1.85	1.87	1.74	1.83	1.61	1.84	2.5	0.220	0.62	10
1,3-Dichloropropane	1.75	1.88	1.47	1.77	1.80	1.84	1.64	1.79	1.57	1.49	1.70	2.5	0.147	0.42	10
2-Hexanone	1.66	1.82	0.88	1.51	1.81	1.34	1.20	1.95	1.03	0.91	1.41	2.5	0.397	1.12	10
Dibromochloromethane	1.55	1.77	1.40	1.58	1.60	1.53	1.45	1.56	1.38	1.31	1.51	2.5	0.132	0.37	10
1,2-Dibromoethane	1.69	1.92	1.40	1.71	1.81	1.83	1.60	1.80	1.54	1.40	1.67	2.5	0.181	0.51	10
Chlorobenzene	1.88	2.24	2.07	2.14	2.11	2.07	2.08	2.01	2.04	1.90	2.05	2.5	0.107	0.30	10
1-Chlorohexane	*	2.67	2.45	2.48	2.47	2.44	2.34	2.35	2.29	*	2.44	2.5	0.117	0.35	10
1,1,1,2-Tetrachloroethane	1.73	2.02	1.79	1.90	1.89	1.81	1.79	1.80	1.81	1.68	1.82	2.5	0.095	0.27	10
Ethylbenzene	1.32	1.83	1.75	1.67	1.61	1.59	1.56	1.58	1.58	1.45	1.59	2.5	0.143	0.40	10
m,p-Xylene	2.92	3.99	3.84	3.57	3.54	3.38	3.38	3.63	3.39	3.06	3.47	5.0	0.323	0.91	20
o-Xylene	1.44	1.87	1.75	1.70	1.70	1.66	1.61	1.79	1.63	1.52	1.67	2.5	0.126	0.36	10
Styrene	1.46	1.82	1.59	1.65	1.63	1.59	1.58	1.70	1.57	1.47	1.61	2.5	0.105	0.30	10
Bromoform	1.61	1.80	1.26	1.58	1.64	1.51	1.36	1.60	1.27	1.16	1.48	2.5	0.205	0.58	10
Isopropyl benzene	1.37	1.97	1.82	1.77	1.72	1.71	1.68	1.61	1.65	1.49	1.68	2.5	0.167	0.47	10
cis-1,4-dichloro-2-butene	0.41	0.52	0.32	0.43	0.44	0.32	0.35	0.36	0.36	0.26	0.38	2.5	0.074	0.21	10
Bromobenzene	1.75	2.02	1.81	1.85	1.90	1.83	1.81	1.80	1.77	1.69	1.82	2.5	0.090	0.25	10
1,2,3-Trichloropropane	1.37	1.48	0.71	1.30	1.43	1.46	1.11	1.44	0.88	0.85	1.20	2.5	0.292	0.82	10
1,1,2,2-Tetrachloroethane	1.88	2.08	1.30	1.77	1.99	2.00	1.59	2.06	1.42	1.34	1.74	2.5	0.307	0.87	10
trans-1,4-Dichloro-2-butene	0.38	0.48	0.24	0.40	0.46	0.27	0.31	0.47	0.26	0.20	0.35	2.5	0.104	0.29	10
n-Propyl benzene	1.60	2.21	2.16	2.13	2.03	2.03	1.98	1.93	1.99	1.80	1.99	2.5	0.180	0.51	10
2-Chlorotoluene	1.59	2.11	1.96	1.94	1.87	1.83	1.87	1.80	1.89	1.81	1.87	2.5	0.133	0.38	10
4-Chlorotoluene	1.73	2.08	1.99	1.97	1.93	1.96	1.89	1.87	1.97	1.77	1.92	2.5	0.105	0.30	10
1,3,5-Trimethyl benzene	1.57	2.11	2.01	1.94	1.96	1.90	1.88	1.82	1.93	1.75	1.89	2.5	0.149	0.42	10
Pentachloroethane	1.57	1.97	1.73	1.70	1.65	1.25	1.58	1.69	1.61	1.56	1.63	2.5	0.180	0.51	10
tert-butyl Benzene	1.60	2.23	2.05	1.99	1.95	1.88	1.95	1.86	1.92	1.78	1.92	2.5	0.166	0.47	10
1,2,4-Trimethyl benzene	1.67	2.16	2.02	2.01	1.96	1.91	1.90	2.03	1.94	1.80	1.94	2.5	0.135	0.38	10
sec-butyl Benzene	1.50	2.26	2.14	2.04	2.02	1.95	1.99	1.85	1.96	1.79	1.95	2.5	0.207	0.58	10
1,3-Dichlorobenzene	1.91	2.25	2.07	2.14	2.08	2.03	2.03	2.00	2.08	1.94	2.05	2.5	0.097	0.27	10
1,4-Dichlorobenzene	2.10	2.48	2.25	2.38	2.22	2.25	2.30	2.14	2.24	2.17	2.25	2.5	0.113	0.32	10
p-Isopropyl toluene	1.87	2.56	2.43	2.50	2.38	2.34	2.42	2.27	2.34	2.14	2.33	2.5	0.198	0.56	10
Benzyl chloride	1.02	1.11	0.73	0.92	0.95	0.40	0.75	0.90	0.60	0.59	0.80	2.5	0.222	0.63	10
1,2-Dichlorobenzene	1.92	2.17	1.92	2.00	2.04	1.91	1.99	1.98	1.94	1.84	1.97	2.5	0.090	0.25	10
n-Butyl benzene	1.33	1.84	1.69	1.72	1.69	1.54	1.65	1.57	1.55	1.42	1.60	2.5	0.150	0.42	10
1,2-Diethylbenzene	1.64	2.12	2.00	1.99	1.96	1.86	1.92	1.81	1.92	1.77	1.90	2.5	0.135	0.38	10
1,2-Dibromo-3-chloropropane	1.30	1.50	0.82	1.23	1.47	1.15	0.98	1.44	0.86	0.86	1.16	2.5	0.267	0.75	10
1,2,4-trichlorobenzene	0.10	0.09	0.12	0.11	0.18	0.08	0.15	0.09	0.07	0.06	0.11	2.5	0.037	0.10	10
Hexachlorobutadiene	1.39	2.11	2.01	1.97	1.84	1.70	1.69	1.64	1.69	1.58	1.76	2.5	0.219	0.62	10
Naphthalene	0.26	0.24	0.20	0.23	0.23	0.03	0.18	0.33	0.17	0.14	0.20	2.5	0.080	0.23	10
1,2,3-Trichlorobenzene	0.05	0.06	0.08	0.06	0.06	0.10	0.06	0.04	0.05	0.04	0.06	2.5	0.018	0.05	10
1,2-Dichloroethene (total)	2.96	4.10	3.86	3.82	3.71	3.62	3.59	3.42	3.47	3.27	3.58	5.0	0.324	0.91	20
Xylene (total)	4.35	5.85	5.58	5.26	5.24	5.04	4.99	5.41	5.02	4.58	5.13	7.5	0.445	1.25	10

Attachment 4

COMPUCHEM a division of Liberty Analytical Corp DATE ___/___/___ INITIAL TIME OF TUNE__________ SHIFT/S(A)_____(B)_____(C)____

GC/MS VOLATILE RUN LOG TIME TUNE EXPIRES__________ LINKER /METHOD__________

COMPUCHEM LOGBOOK 11 X 17 (F50052)

PREVENTIVE MAINTENANCE______________________________

[illegible]	[illegible]	[illegible]	[illegible]	DATE	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
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SUPERVISOR APPROVAL______________________________ Date____________

Tune (ID #7008) Lot No. ____________________

Calibration Group Code / Lot No. ________________

The presence of the Chemist's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist together with the chemist's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

5/30/02;doc

Attachment 5

Analyst Capability Study

Laboratory Name/North Carolina Certificate Number: CompuChem/79
Analyst: Jeremy Smith
Study Date: April 18, 2000
Method: 5035/8260B, 5gm
Soil
Instrument/Column/Detector: f50052

Compounds	TrueVal ug/kg	Rep #1 ug/kg	Rep #2 ug/kg	Rep #3 ug/kg	Rep #4 ug/kg	Mean ug/kg	Mean % R	EPA %R	SD(n-1) ug/kg	EPA %RSD	-3SD of (x)	+3SD of (x)	-3SD % R	+3SD %R	RSD %
Dibromofluoromethane	50	55.16	54.72	50.24	54.07	53.55	107	NA	2.25	NA	46.8	60.3	87	113	4.2
1,2-Dichloroethane-d4	50	55.39	56.72	52.84	54.28	54.81	110	NA	1.65	NA	49.9	59.8	91	109	3.0
Toluene-d8	50	48.85	49.86	45.84	48.42	48.24	96	NA	1.71	NA	43.1	53.4	89	111	3.5
Bromofluorobenzene	50	48.68	48.32	42.59	45.19	46.20	92	NA	2.87	NA	37.6	54.8	81	119	6.2
Chloromethane	50	48.03	46.52	41.81	45.74	45.53	91	NA	2.65	NA	37.6	53.5	83	117	5.8
Vinyl Chloride	50	46.69	44.65	38.97	44.32	43.66	87	34.2	3.30	13.0	33.8	53.5	77	123	7.5
Bromomethane	50	53.44	53.89	47.79	51.74	51.72	103	NA	2.78	NA	43.4	60.0	84	116	5.4
Chloroethane	50	60.40	59.08	53.29	58.47	57.81	116	NA	3.12	NA	48.5	67.2	84	116	5.4
1,1-Dichloroethene	50	53.53	55.28	48.78	52.65	52.56	105	79.2	2.75	5.7	44.3	60.8	84	116	5.2
Acetone	130	145.6	145.1	149.7	147.5	147.0	113	NA	2.08	NA	140.7	153.2	96	104	1.4
Carbon disulfide	50	53.52	55.46	49.62	53.18	52.95	106	NA	2.43	NA	45.6	60.2	86	114	4.6
Methylene Chloride	50	59.07	57.92	51.39	56.62	56.25	113	107	3.39	9.1	46.1	66.4	82	118	6.0
trans-1,2-Dichloroethene	50	54.97	55.73	49.14	53.05	53.22	106	104	2.95	0.7	44.4	62.1	83	117	5.5
Methyl tert butyl ether	50	58.46	59.68	54.58	56.71	57.36	115	NA	2.22	NA	50.7	64.0	88	112	3.9
1,1-Dichloroethane	50	51.63	54.30	47.29	50.78	51.00	102	84.4	2.89	6.4	42.3	59.7	83	117	5.7
cis-1,2-Dichloroethene	50	54.79	57.23	49.56	52.02	53.40	107	113	3.33	9	43.4	63.4	81	119	6.2
2-Butanone	130	128.5	124.5	126.4	132.0	127.9	98	NA	3.21	NA	118.3	137.5	92	108	2.5
Chloroform	50	55.08	58.27	51.15	54.26	54.69	109	116	2.93	12.2	45.9	63.5	84	116	5.4
1,1,1-Trichloroethane	50	51.59	53.51	47.34	51.33	50.94	102	117	2.59	21.2	43.2	58.7	85	115	5.1
Carbon tetrachloride	50	56.22	59.49	53.77	58.58	57.02	114	112	2.56	9.4	49.3	64.7	87	113	4.5
1,2-Dichloroethane	50	56.49	57.33	52.43	56.12	55.59	111	NA	2.17	NA	49.1	62.1	88	112	3.9
Benzene	50	49.77	52.32	44.86	48.70	48.91	98	103	3.10	11.2	39.6	58.2	81	119	6.3
Trichloroethene	50	53.47	55.09	48.43	50.17	51.79	104	94.6	3.03	12.7	42.7	60.9	82	118	5.9
1,2-Dichloropropane	50	51.36	52.93	46.55	48.84	49.92	100	117	2.81	10.5	41.5	58.3	83	117	5.6
Bromodichloromethane	50	56.84	59.52	52.46	55.27	56.02	112	117	2.95	13.1	47.2	64.9	84	116	5.3
cis-1,3-Dichloropropene	50	54.47	57.15	48.88	50.81	52.83	106	NA	3.70	NA	41.7	63.9	79	121	7.0
4-Methyl-2-pentanone	130	122.1	116.4	120.0	127.6	121.5	93	NA	4.68	NA	107.5	135.6	88	112	3.9
Toluene	50	48.19	49.79	45.38	48.02	47.85	96	118	1.83	16.9	42.4	53.3	89	111	3.8
trans-1,3-Dichloropropene	50	53.69	54.96	51.11	52.83	53.15	106	NA	1.62	NA	48.3	58.0	91	109	3.0
1,1,2-Trichloroethane	50	50.80	51.03	48.82	54.06	51.18	102	111	2.16	12.1	44.7	57.7	87	113	4.2
Tetrachloroethene	50	51.16	52.03	48.45	51.95	50.90	102	NA	1.68	NA	45.9	55.9	90	110	3.3
2-Hexanone	130	120.6	119.6	119.7	122.5	120.6	93	NA	1.36	NA	116.5	124.7	97	103	1.1
Dibromochloromethane	50	56.73	57.40	52.38	56.46	55.74	111	118	2.28	12.5	48.9	62.6	88	112	4.1
Chlorobenzene	50	52.07	52.48	47.87	49.63	50.51	101	99.3	2.16	15	44.0	57.0	87	113	4.3
Ethylbenzene	50	50.75	52.58	46.32	47.34	49.25	98	112	2.92	17.5	40.5	58.0	82	118	5.9
m,p-Xylene	100	97.59	104.76	90.44	94.19	96.75	97	98.5	6.09	15.7	78.5	115.0	81	119	6.3
o-Xylene	50	50.15	52.09	45.95	46.33	48.63	97	103	2.99	17.3	39.7	57.6	82	118	6.1
Styrene	50	48.39	52.51	44.56	45.84	47.83	96	101	3.51	15.7	37.3	58.3	78	122	7.3
Bromoform	50	57.64	60.05	56.53	61.67	58.97	118	122	2.32	9.9	52.0	65.9	88	112	3.9
1,1,1,2-Tetrachloroethane	50	55.25	53.05	49.84	57.87	54.00	108	NA	3.40	NA	43.8	64.2	81	119	6.3
1,2-Dichloroethene (total)	100	109.8	112.6	98.6	105.3	106.6	107	NA	6.11	NA	88.2	124.9	83	117	5.7
Xylene (total)	150	161.7	171.8	149.3	154.0	159.2	106	NA	9.84	NA	129.7	188.7	81	119	6.2

CompuChem
a division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure ______ revised procedure √ outdated procedure (archive) ______

- Procedure Code: SPP-238 SOP Section #: 1.1.4.1 Revision #: 3

SOP Title: Effective date: (QA fills in)

Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap Using SW846 Method 5035

9/23/02

- Procedure prepared by: Diane C. Ellmore Date: 9/23/02
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 9/23/02
- Reason for change: Annual Review
- This procedure meets the requirements of the following approved method references:

New York State Analytical Services Protocol (NYSASP), June 2000, plus revisions; NELAC Standards, June 2000, plus revisions; SW-846, 3rd Edition, Update III, Method 5035; US EPA CLP SOW OLM04.2, OLM04.3, plus revisions

Procedure approved by Quality Assurance Representative: Date:
(Not needed if signed above)

ORIGINAL

Effective 1-1-96, on an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: ____________ Date: ______

Annual Review—Signature: ____________ Date: ______

Annual Review—Signature: ____________ Date: ______

Sample Preparation Procedure -238: Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap using SW846 Method 5035

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Sample Preparation Procedure -238: Preparation of Soil/Sediment/Sludge Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap using SW846 Method 5035

1.0 Scope and Application

This procedure is used to prepare soil, sediment, and sludge samples for the analysis of volatile organic compounds by the closed-system purge and trap process using GC/MS Method 8260B or CLP. The procedure is based on Method 5035. The prepared sample may also be analyzed for GRO by Method 8015B. Provisions are also included to prepare (and preserve) samples with methanol when higher concentrations of volatiles are present in the sample.

Method detection limits and reporting limits are found in the respective analytical SOPs for the methods described in the above paragraph.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A five (5) gram sample is taken in the field using a disposable EnCore sampling device. At the laboratory and within forty-eight (48) hours of sampling, the sample is weighed into a standard forty (40) milliliter volatile vial containing a magnetic stirring bar and five (5) mL of a sodium bisulfate solution (0.1 g of sodium bisulfate per gram of sample weighed.) The vial is sealed with a screw-top, PTFE-lined, septum-sealed cap. The sample container is stored at 4°C ± 2°C until analysis, which must be completed within 14 days of collection.

For screening purposes, and also to provide a mechanism to determine the concentration of target analytes which may be present at high levels, the contents of another 5 gram EnCore sampler are also weighed and placed into a vial containing methanol.
For some projects, a 25 gram EnCore sampler may be used and its contents are placed in a 2 oz. jar and preserved with methanol.

Depending on specific project requirements, samples may be received by the laboratory already in sealed 40 mL vials, containing a stirring bar and sodium bisulfate solution, or preserved with methanol.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

For CLP the reporting limit is the Contract Required Quantitation Limit (CRQL) for organics.

3.3 Reporting Units – µg/Kg

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days **if requested by the client**) beginning with the receipt of the first sample.

NOTE: The Army Corps of Engineers does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

3.5 GRO – Gasoline Range Organics

4.0 Interferences

4.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The an_lytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks.

4.2 The use of non-PTFE plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging devise must be avoided.

4.3 Samples can be contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. A trip blank carried through sampling and storage serves as a check of contamination.

4.4 The laboratory where volatiles are prepared and analyzed should be completely free of solvents. Persons leaving the organic sample preparation laboratory must not enter the volatile laboratory until sufficient time has passed to avoid the introduction of solvent vapors.

5.0 Safety

5.1 When following this procedure, be sure to wear protective gloves, lab coats, safety glasses and work under a hood.

5.2 Laboratory staff are encouraged to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment & Supplies

6.1 Sample containers - standard forty (40) milliliter volatile vial with screw-top, PTFE-lined, septum-sealed cap. **Vials are purchased pre-cleaned from certifying vendors.**

6.2 Magnetic stirring bar - PTFE or glass coated .

6.3 Stainless steel spatulas.

6.4 Top-loading balance, capable of weighing accurately to 0.01 g

6.5 Five (5) or twenty-five (25) gram disposable EnCore sampling device.

6.6 Graduated pipettes capable of delivering 1-10 mL.

7.0 Reagents & Standards

Standard preparation are contained in the standard operating procedures (SOP) for that area (Section 7.0 of the SOP collection.)

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remained of this SOP as DI water.

7.2 Methanol – purge and trap grade or equivalent.

7.3 Sodium bisulfate (NaHSO4) ACS reagent grade.

7.3.1 20% sodium bisulfate solution – 200 g $NaHSO_4$ in 1000 ml DI water.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

9.0 Quality Control

9.1 A method blank is prepared for every preparation batch following steps 11.3 - 11.5, and consists of a purified solid matrix, i.e. Ottawa sand.

9.2 Steps 11.3 - 11.5 are repeated twice more from a designated sample and used for the matrix spike (MS) and matrix spike duplicate (MSD). An additional 5-g portion of a purified solid matrix (Ottawa sand) is weighed into a separate sample container for the blank spike (BS). This is called a matrix spike blank for the NYSASP. A BS is prepared with each preparation batch.

9.3 Duplicates, at a frequency of 10%, are required when processing samples submitted to meet the regulatory requirements of North Carolina.

10.0 Calibration & Standardization

10.1 Balance Calibration

10.1.1 Ensure the balance is calibrated for the day prior to weighing samples. Refer to **Quality Control** SOP 13.16, "Top Loading Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Glassware must be scrupulously clean. The glassware and magnetic stir bars are washed with hot soapy water, rinsed with hot water and finally rinsed with laboratory pure water. The glassware is dried at 105 ± 5°C for one (1) hour or overnight in an oven. If the glassware has been stored in the laboratory environment, place it in oven for one (1) hour (105 ± 5°C) and allow it to cool in a contaminant free environment before using. Alternatively, rinse thoroughly with DI water before use.

The 40 mL VOA vials are purchased pre-cleaned **by certifying vendors**.

11.2 When the samples have come to room temperature and are ready to be prepared, assemble the designated materials in the hood.

11.3 For each sample, label (with permanent ink **typically** on white tape) three (3) standard volatile vial sample containers with the laboratory ID number of the sample.

11.4 Open the five (5) gram disposable EnCore sampling device. Do not discard any supernatant liquid.

11.5 Place a vial (with cap), containing a magnetic stirring bar, on the top loading balance, and add a 5 ml aliquot of $NaHSO_4$ solution (7.3.1). **The weight is recorded on the EnCore Preparation Worksheet (attachment 1).** Add the contents of the EnCore sampler to the vial. Read and record weight. Seal the volatile vial with the PTFE-lined screw cap.

11.6 Prepare a back-up aliquot by repeating step 11.4 and 11.5.

11.7 Place another vial, containing a magnetic stirring bar, on the top-loading balance, add 0.2g ± 0.1g of sodium bisulfate, and 5 ml of organic-free water. Record weight. Add 1.0g ± 0.1g of the sample contained in bottle submitted for percent moisture. Read and record weight. Seal the vial with the PTFE-lined screw cap and record the weight. This is used for those analytes that exceed the instrument calibration range in the 5g analysis.

Note: This is an optional step and may be performed after the initial analysis of a 5 gram sample.

11.8 Place another 40 ml vial and cap on the top-loading balance and transfer 5.0 ml ± 0.1 ml of methanol. Record the weight. Transfer the contents of a 5 gram EnCore sampler to the vial. Seal the vial with the PTFE-lined screw cap and record the weight. Use this sample for screening purposes and medium/high level analysis, if necessary.

11.9 Repeat steps 11.4-11.8 until the required number of samples are prepared, completing the worksheet as the preparation is accomplished.

11.10 If, during the addition of a sample to a vial containing the sodium bisulfate solution, effervescing occurs, that sample should be properly discarded. Another vial (with stirring bar) should be obtained, weighed, the contents of a 5 gram EnCore sample added and the vial should be sealed with a PTFE-lined screw cap and the weight recorded. This sample should be placed in a freezer at −12°C ± 2°C. Pre-testing for effervescence can also be performed using the bottle supplied for percent moisture.

11.12 Samples prepared by this method are now ready for analysis by the appropriate analytical method. When the set is complete, store the samples in the appropriate volatile GC or GC/MS laboratory refrigerator until analysis. The refrigerator temperature must be at 2°-4.4°C in order to meet NCDENR storage requirements.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst (Attachment 2). The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP Statement of Work for Organics, Multi-Media, Multi-Concentration, OLM04.2, **OLM04.3,** plus revisions

16.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96, Method 5035, Method 8260B

16.3 Standard Methods for the Examination of Water and Wastewater, 18th Edition (1992), 19th (1995) and 20th (1998) Editions upon promulation, Method 1080

16.4 New York State Analytical Services Protocol (NYSASP), June, 2000, **plus revisions**

16.5 QCSOP: Proper Documentation Procedures

16.6 QCSOP: Numerical Data Reduction

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, **June 2000**, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 New York State Environmental Laboratory Approval Program, Certification Manual, October 15, 1999, plus revisions.

16.12 CompuChem Quality Manual, Revision 2, 10/26/01, plus revisions

16.13 Sample Preparation Procedure –143, "% Moisture Determination (Undecanted) (EPA CLP SOW, SW8746, and NYSASP", SOP Section 2.8.1.

16.14 Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples."

16.15 **Quality Control** SOP 13.16, "Top Loading Balance Calibration and Maintenance"

17.0 <u>Attachments as Tables, Diagrams, Flowcharts & Validation Data</u>

17.1 Attachment 1 – EnCore Preparation Worksheet

17.2 Attachment 2 – Single Analyst Capability Study

Attachment 1

COMPUCHEM

a Division of Liberty Analytical

EnCore™ Preparation Worksheet

-238 / -243 / -233

SDG:________________ Prepared By:__________________________ Date:__________________

	LoginNum	Date	Time	Weight of Jar (g)	Weight of Jar + Sample (g)	Weight of Sample (g)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						

Volume of Methanol Added:____________________

Lot Numbers of Preservatives Sodium Bisulfate:__________________ Methanol:______________

Attachment 2

Laboratory Name/North Carolina Certificate Number: CompuChem/79
Analyst: Jeremy Smith
Study Date: April 18, 2000
Method: 5035/8260B, 5gm Soil
Instrument/Column/Detector:
f50052

Compounds	TrueVal ug/kg	Rep #1 ug/kg	Rep #2 ug/kg	Rep #3 ug/kg	Rep #4 ug/kg	Mean ug/kg	Mean % R	EPA %R	SD(n-1) ug/kg	EPA %RSD	-3SD of (x)	+3SD of (x)	-3SD % R	+3SD %R	RSD %
Dibromofluoromethane	50	55.16	54.72	50.24	54.07	53.55	107	NA	2.25	NA	46.8	60.3	87	113	4.2
1,2-Dichloroethane-d4	50	55.39	56.72	52.84	54.28	54.81	110	NA	1.65	NA	49.9	59.8	91	109	3.0
Toluene-d8	50	48.85	49.86	45.84	48.42	48.24	96	NA	1.71	NA	43.1	53.4	89	111	3.5
Bromofluorobenzene	50	48.68	48.32	42.59	45.19	46.20	92	NA	2.87	NA	37.6	54.8	81	119	6.2
Chloromethane	50	48.03	46.52	41.81	45.74	45.53	91	NA	2.65	NA	37.6	53.5	83	117	5.8
Vinyl Chloride	50	46.69	44.65	38.97	44.32	43.66	87	34.2	3.30	13.0	33.8	53.5	77	123	7.5
Bromomethane	50	53.44	53.89	47.79	51.74	51.72	103	NA	2.78	NA	43.4	60.0	84	116	5.4
Chloroethane	50	60.40	59.08	53.29	58.47	57.81	116	NA	3.12	NA	48.5	67.2	84	116	5.4
1,1-Dichloroethene	50	53.53	55.28	48.78	52.65	52.56	105	79.2	2.75	5.7	44.3	60.8	84	116	5.2
Acetone	130	145.6	145.1	149.7	147.5	147.0	113	NA	2.08	NA	140.7	153.2	96	104	1.4
Carbon disulfide	50	53.52	55.46	49.62	53.18	52.95	106	NA	2.43	NA	45.6	60.2	86	114	4.6
Methylene Chloride	50	59.07	57.92	51.39	56.62	56.25	113	107	3.39	9.1	46.1	66.4	82	118	6.0
trans-1,2-Dichloroethene	50	54.97	55.73	49.14	53.05	53.22	106	104	2.95	0.7	44.4	62.1	83	117	5.5
Methyl tert butyl ether	50	58.46	59.68	54.58	56.71	57.36	115	NA	2.22	NA	50.7	64.0	88	112	3.9
1,1-Dichloroethane	50	51.63	54.30	47.29	50.78	51.00	102	84.4	2.89	6.4	42.3	59.7	83	117	5.7
cis-1,2-Dichloroethene	50	54.79	57.23	49.56	52.02	53.40	107	113	3.33	9	43.4	63.4	81	119	6.2
2-Butanone	130	128.5	124.5	126.4	132.0	127.9	98	NA	3.21	NA	118.3	137.5	92	108	2.5
Chloroform	50	55.08	58.27	51.15	54.26	54.69	109	116	2.93	12.2	45.9	63.5	84	116	5.4
1,1,1-Trichloroethane	50	51.59	53.51	47.34	51.33	50.94	102	117	2.59	21.2	43.2	58.7	85	115	5.1
Carbon tetrachloride	50	56.22	59.49	53.77	58.58	57.02	114	112	2.56	9.4	49.3	64.7	87	113	4.5
1,2-Dichloroethane	50	56.49	57.33	52.43	56.12	55.59	111	NA	2.17	NA	49.1	62.1	88	112	3.9
Benzene	50	49.77	52.32	44.86	48.70	48.91	98	103	3.10	11.2	39.6	58.2	81	119	6.3
Trichloroethene	50	53.47	55.09	48.43	50.17	51.79	104	94.6	3.03	12.7	42.7	60.9	82	118	5.9
1,2-Dichloropropane	50	51.36	52.93	46.55	48.84	49.92	100	117	2.81	10.5	41.5	58.3	83	117	5.6
Bromodichloromethane	50	56.84	59.52	52.46	55.27	56.02	112	117	2.95	13.1	47.2	64.9	84	116	5.3
cis-1,3-Dichloropropene	50	54.47	57.15	48.88	50.81	52.83	106	NA	3.70	NA	41.7	63.9	79	121	7.0
4-Methyl-2-pentanone	130	122.1	116.4	120.0	127.6	121.5	93	NA	4.68	NA	107.5	135.6	88	112	3.9
Toluene	50	48.19	49.79	45.38	48.02	47.85	96	118	1.83	16.9	42.4	53.3	89	111	3.8
trans-1,3-Dichloropropene	50	53.69	54.96	51.11	52.83	53.15	106	NA	1.62	NA	48.3	58.0	91	109	3.0
1,1,2-Trichloroethane	50	50.80	51.03	48.82	54.06	51.18	102	111	2.16	12.1	44.7	57.7	87	113	4.2
Tetrachloroethene	50	51.16	52.03	48.45	51.95	50.90	102	NA	1.68	NA	45.9	55.9	90	110	3.3
2-Hexanone	130	120.6	119.6	119.7	122.5	120.6	93	NA	1.36	NA	116.5	124.7	97	103	1.1
Dibromochloromethane	50	56.73	57.40	52.38	56.46	55.74	111	118	2.28	12.5	48.9	62.6	88	112	4.1
Chlorobenzene	50	52.07	52.48	47.87	49.63	50.51	101	99.3	2.16	15	44.0	57.0	87	113	4.3
Ethylbenzene	50	50.75	52.58	46.32	47.34	49.25	98	112	2.92	17.5	40.5	58.0	82	118	5.9
m,p-Xylene	100	97.59	104.76	90.44	94.19	96.75	97	98.5	6.09	15.7	78.5	115.0	81	119	6.3
o-Xylene	50	50.15	52.09	45.95	46.33	48.63	97	103	2.99	17.3	39.7	57.6	82	118	6.1
Styrene	50	48.39	52.51	44.56	45.84	47.83	96	101	3.51	15.7	37.3	58.3	78	122	7.3
Bromoform	50	57.64	60.05	56.53	61.67	58.97	118	122	2.32	9.9	52.0	65.9	88	112	3.9
1,1,1,2-Tetrachloroethane	50	55.25	53.05	49.84	57.87	54.00	108	NA	3.40	NA	43.8	64.2	81	119	6.3
1,2-Dichloroethene (total)	100	109.8	112.6	98.6	105.3	106.6	107	NA	6.11	NA	88.2	124.9	83	117	5.7
Xylene (total)	150	161.7	171.8	149.3	154.0	159.2	106	NA	9.84	NA	129.7	188.7	81	119	6.2